

HUNTERS POINT ANNEX  
TREASURE ISLAND NAVAL STATION  
PCB VERIFICATION SAMPLING RESULTS

AREA 2

Prepared for Western Division Naval  
Facilities, Engineering Command

December 1987

Prepared by  
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HUNTERS POINT ANNEX  
TREASURE ISLAND NAVAL STATION

AREA 2 - VERIFICATION SOIL SAMPLING SUMMARY  
FOR CLEANUP OF PCB CONTAMINATED SOILS NEAR FORMER BUILDING 503

This report summarizes the Area 2 verification sampling results for the second phase of the interim cleanup of Polychlorinated Biphenyl (PCB) contaminated soils near former Building 503 at Hunters Point Annex. Area 1 verification sampling results for Phase I of the interim cleanup were summarized in references 1 and 2. Upon completions of Phase III activities in Area 3, a final report will be prepared that summarizes verification sampling results for all phases of the project.

PROJECT ORGANIZATION AND RESPONSIBILITY

Four groups are directly involved in the cleanup of the site: Navy, excavation and disposal contractor, sample verification consultant, and an analytical laboratory. The PCB cleanup project is directed by the Western Division Naval Facilities Engineering Command (WESTDIV), San Bruno, California. WESTDIV organizes, contracts, and coordinates the cleanup work with contractors and consultants; communicates with the regulatory agencies; inspects the site; and tracks work progress.

The cleanup contractor is American Environmental Management Corp., Oakland, California. The contractor is responsible for excavation and removal of soil from the PCB site, site containment, and health and safety of American Environmental personnel.

Sampling of the site is performed by ERM-WEST, Walnut Creek, California. ERM-WEST is responsible for sampling the site and reporting the PCB results to the Navy; setting-up the verification

sampling grid; overseeing health and safety of ERM-West personnel; and reporting sampling results.

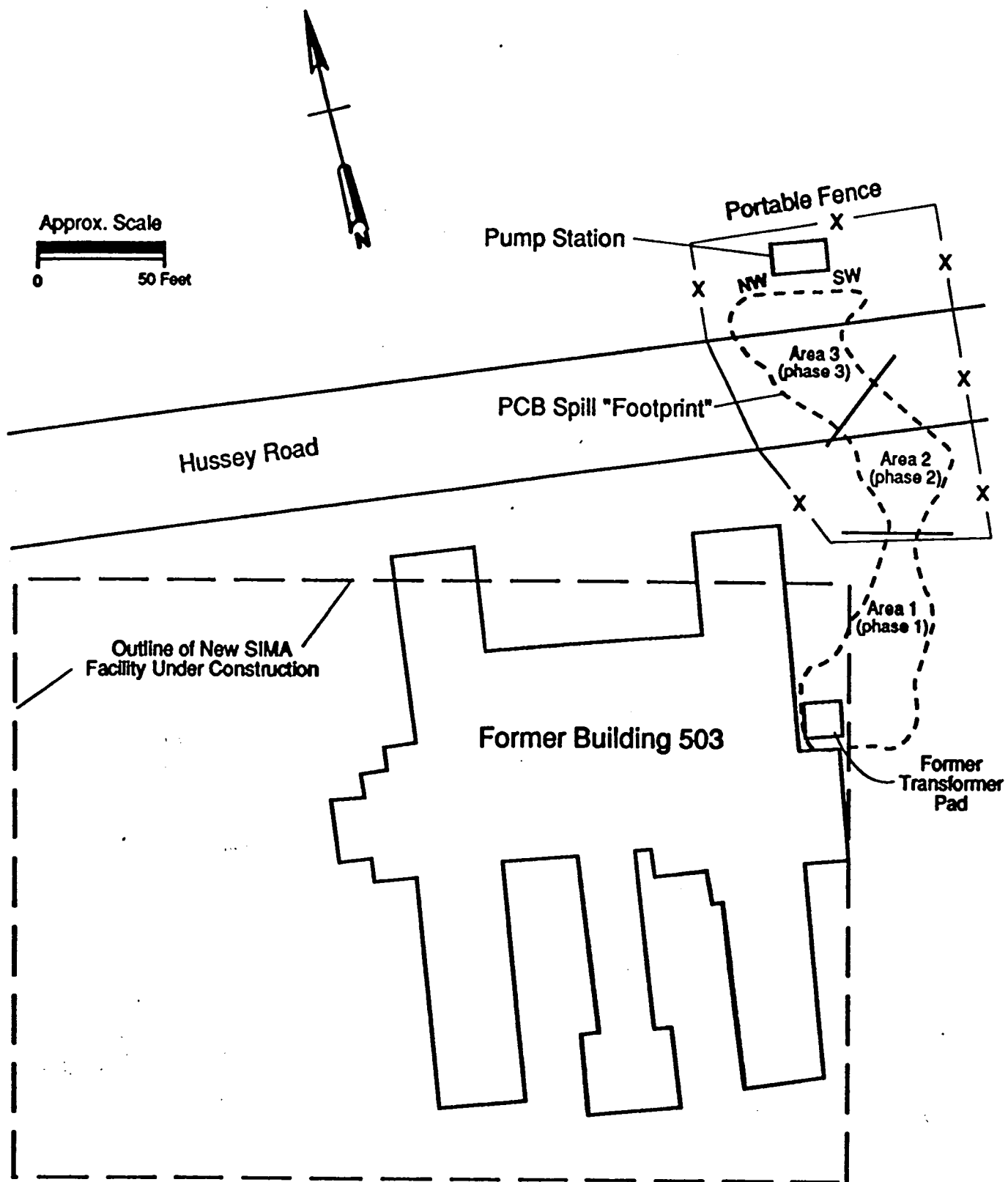
Central Coast Analytical Services, San Luis Obispo, California is the laboratory used to perform the PCB analytical procedures. Central Coast Analytical Services is a certified hazardous waste laboratory and the laboratory used during the Area 1 cleanup phase. (References 1 and 2)

#### VERIFICATION SAMPLING PROGRAM

Contaminated soils in Area 2, as shown in Figure 2-1, were identified, excavated, and removed by American Environmental Management Corp. Contaminated soils were manifested and disposed at a Class 1 landfill.

The decision to verify the soil clean-up work in Area 2, through the development and preparation of a formal verification sampling plan, was based on field samples taken and analyzed by a McGraw Edison field PCB kit. The correlation between field sample results and certified laboratory reports has been confirmed and previously discussed and reported in both the Interim Report and addendum (Reference 1 and 2). Experience and comparison of split samples analyzed concurrently by the field test kit and a certified analytical laboratory consistently indicated that the field kit was conservatively showing PCB concentrations higher than the certified laboratory. The field test kit was therefore used as a screening device to help direct field activities.

The following section summarizes the verification sampling program for Area 2. The agreed interim cleanup level for this site is 25 mg/kg of PCB. Detailed laboratory reports for the verification sample analysis are presented in Appendix A. U.S. Environmental Protection Agency (USEPA) protocol was used to design the verification sampling program (Reference 3).



**Figure 2-1**  
**PCB Site-Work Areas**

## Sample Layout Procedures

The layout of the verification sampling program for Area 2 is shown on Figure 2-2. The location of sampling points was based on USEPA protocol for verification sampling (Reference 3, EPA manual on "Verification of PCB Spill Cleanup by Sampling and Analysis", Interim Report No. 2, August, 1985). Each point was spaced at 11.25 foot intervals. This spacing was based on a spill radius of 37.5 feet (longest length of Area 2 was 75 feet), a sample size of 37 (reference Table 4 of the above manual), and a sample spacing based on 0.3 times the radius of the spill area (Table 2 of above manual).

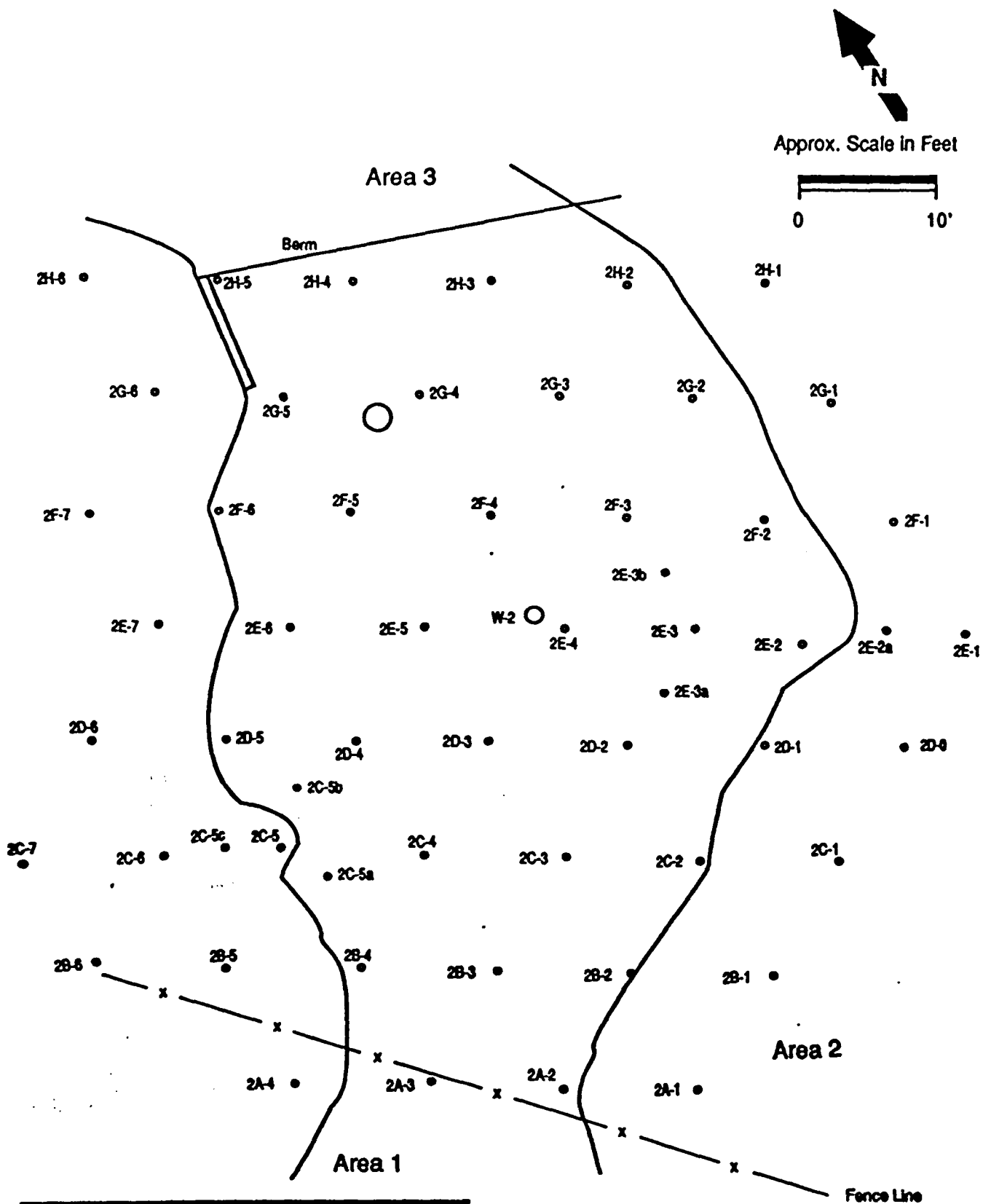
The number of sample grid points established in the field exceeded the 37 sample size referenced above. Additional grid points were identified on the outside periphery of the excavation to confirm that the extent of the surface PCB contamination had been accurately identified. The total number of grid points sampled and analyzed was 50.

As indicated on Figure 2-2, three of the sample points have been triangulated and located relative to the existing pumping station. These three points will allow re-establishment of the sampling grid at a future time, if necessary.

## Sampling and Sample Tracking

The sampling procedure and sample tracking was coordinated with the Department of Health Services (DHS). The grid points were initially sampled on September 3 and 4, 1987. After results of the September 3-4, 1987 samples were reported, areas where soil contamination exceeded the cleanup criteria of 25 ppm PCBs were excavated by American Environmental. A second round of verification sampling was performed on October 16, 1987 after additional soil excavation.

At the time of sampling, a representative from the DHS was



Distance (ft) to Pumping Station		
Grid Point	NW Corner *	SW Corner *
2C-1	107	97.5
2C-6	98.5	99
2H-1	68	56

**Figure 2-2**  
**Area 2 Verification Sampling Grid**

\* See Figure 2-1 for Location

present on site and retained seven samples, selected at random, from the September 3-4, 1987 program (2A-1, 2C-2, 2C-5, 2C-6, 2E-1, 2E-4, and 2G-4) and two samples (2C-5 and 2E-3) from the October 16, 1987 program. These samples were taken to the DHS laboratory for sample preparation (air drying), splitting, and subsequent forwarding of one-half of each sample to Central Coast Analytical Services for PCB analysis by EPA Method 8080.

To track samples from the field to the laboratory, chain of custody documentation was prepared and sealed with the field-to-laboratory transport container. Chain of custody forms prepared and used during the sampling program are presented in Appendix A.

September 3-4, 1987 Sampling. On September 3, 1987 the periphery of the site, outside the excavation, was sampled using a hollow stem drill rig. The drill rig was used to break through the asphalt paving and to push a steel or brass tube for retrieval of undisturbed samples at depths varying between 1.33 and 3.0 feet. The depth of sample was previously confirmed with the DHS before drilling and follows the procedures established during the Area 1 sampling program. After sample retrieval, sample tubes were covered with teflon and capped prior to icing and transporting to the laboratory.

The interior of the excavation was sampled on September 4, 1987. In some areas, ground water was pumped from the excavation before sampling. At each grid point, a six ounce surface soil sample was taken with a wooden disposable tongue depressor and placed in a glass jar; depth of sample was approximately two centimeters. Depth of the excavation ranged from one foot to approximately six feet. During the sampling program, two field blanks were taken and analyzed by the laboratory.

October 16, 1987 Sampling. The September sampling program identified areas that did not meet cleanup levels. After additional excavation of these specific areas, surface samples were taken in the re-excavated areas. Sampling procedures were

identical to September 3-4, 1987 protocol for surface sampling within the excavated area.

#### ANALYTICAL PROCEDURES

Once the samples were received at the Central Coast Analytical Services laboratory the samples were prepared, extracted, and analyzed for PCBs. Sample preparation and extraction generally followed EPA method 3550 - Sonication Extraction. However, instead of a 1mm X 1mm sieve required by the EPA method, Central Coast used a #10 sieve (2mm X 2mm) to be consistent with sample preparation required by California Administrative Code, Title 22, Division 4, Chapter 30, Section 66700 (c) (1).

At the request of the DHS, the samples received at the laboratory were air dried before analysis for PCBs. This was done after determination of percent moisture in the original sample. Central Coast's detection limits were such that a 20 gram wet weight sample was used and mixed with  $\text{Na}_2\text{SO}_4$  then sonicated. The extraction solvent was 1:1 hexane:acetone (B & J) chromatography grade. The extract sample was filtered and reduced in volume using a Kuderna-Danish concentrator.

PCBs, if present in the sample extract, were identified using EPA method 8080 - Organochlorine Pesticides and PCBs.

Central Coast's "Quality Assurance/Quality Control Procedure" as summarized in Appendix B, requires blanks, duplicates, and spikes to be analyzed once per batch, once per matrix type or once per 20 samples, whichever is more frequent. For this project, however, duplicates were run once per batch, once per matrix type or once per 10 samples analyzed, whichever was more frequent.

## RESULTS

Results of the sampling program are shown in Table 2-1. Two rounds of sampling were performed: the first on September 3-4, 1987 and a second round on October 16, 1987.

### September 3-4, 1987 Results

In Table 2-2 the split samples analyzed by the DHS and Central Coast Analytical Services are compared by grid area for the September 3-4, 1987 sampling program. Using a conservative approach, it was determined that if any one value from either of the laboratories exceeded the cleanup criteria of 25 ppm, then that specific area would be reexcavated and resampled in the subsequent verification sampling program.

After the first round results were received, samples from five grid points (2C-5, 2D-2, 2E-2, 2E-3, and 2E-4) exceeded the 25 ppm cleanup level. All the sample points were within the excavation.

Based on the first round sample results, the soils within and adjacent to the five sample points exceeding the cleanup criteria were excavated and disposed at a class 1 landfill. The additional cleanup area was defined as the area circumscribing the contaminated sample location with the perimeter half way between a "clean" sample grid point and the contaminated location. Depth of additional excavation was determined by field sampling and testing of the soil with the field PCB kit. Approximately 54 cubic yards of soil were removed from the excavation after the September 3-4, 1987 sampling program.

### October 16, 1987 Results

After soil removal, the areas at the five contaminated grid points were resampled to verify that the cleanup criteria had been

TABLE 2-1

HUNTERS POINT ANNEX  
TREASURE ISLAND NAVAL STATION

VERIFICATION SOIL SAMPLING RESULTS  
FROM CENTRAL COAST ANALYTICAL SERVICES  
FOR AREA 2

Sample Description	September 3-4, 1987			October 16, 1987		
	Percent Moisture	PCBs <sup>1</sup> , D.L. <sup>2</sup> , mg/kg	Conc <sup>3</sup>	Percent Moisture	PCBs <sup>1</sup> , D.L. <sup>2</sup> , mg/kg	Conc <sup>3</sup>
2A-1 <sup>4</sup>	6.0	0.2	ND <sup>7</sup>			
2A-2	3.2	0.02	5.0			
2A-3	7.1	0.02	2.2			
2A-4	2.7	0.02	0.61			
2B-1	-- <sup>5</sup>	0.02	<0.02			
2B-2	16	0.02	16			
2B-3	7.2	0.02	0.44			
2B-3 (Dup)	7.1	0.02	--			
2B-4	5.1	0.02	1.0			
2B-5	3.8	0.02	0.60			
2B-6	-- <sup>5</sup>	0.02	<0.02			
2C-1	-- <sup>5</sup>	0.02	0.04			
2C-2 <sup>4</sup>	30	0.02	25			
2C-3	12	0.02	3.2			
2C-4	25	0.02	15			
2C-5 <sup>4,6</sup>	<0.1	0.02	51	--	0.1	1.3
2C-5A				15	0.05	2.4
2C-5A (Dup)				15	0.05	2.7
2C-5A (Tripl)				15	0.05	2.9
2C-5B				21	0.05	11
2C-5C				19	0.05	1.5
2C-6 <sup>4</sup>	4.7	0.02	0.65			
2C-7	-- <sup>5</sup>	0.02	<0.02			
2D-0	-- <sup>5</sup>	0.02	<0.02			
2D-1	30	0.02	3.5			
2D-2	41	0.02	21	24	0.05	16
2D-3	31	0.02	4.3			
2D-4	21	0.02	2.2			
2D-5	1.2	0.02	2.2			
2D-6	-- <sup>5</sup>	0.02	<0.02			

TABLE 2-1 (continued)  
VERIFICATION SOIL SAMPLING RESULTS  
FOR AREA 2

Sample Description	September 3-4, 1987			October 16, 1987		
	Percent Moisture	PCBs <sup>1</sup> , mg/kg D.L. <sup>2</sup>	Conc <sup>3</sup>	Percent Moisture	PCBs <sup>1</sup> , mg/kg D.L. <sup>2</sup>	Conc <sup>3</sup>
2E-1 <sup>4</sup>	5.8	0.2	ND <sup>7</sup>			
2E-1(Dup) <sup>4</sup>	5.8	0.2	ND <sup>7</sup>			
2E-2	41	0.02	36	18	0.05	3.0
2E-2(Dup)				18	0.05	3.0
2E-2A				5	0.05	2.2
2E-3 <sup>6</sup>	25	0.02	59	--	0.05	7.9
2E-3A				20	0.05	15
2E-3B				16	0.05	0.79
2E-4 <sup>4</sup>	22	0.02	24	19	0.05	8.9
2E-4(Dup) <sup>4</sup>	22	0.02	25			
2E-4(Spike)				19	0.05	14
2E-5	20	0.02	4.6			
2E-5(Dup)	20	0.02	4.2			
2E-6	17	0.02	0.6			
2E-7	-- <sup>5</sup>	0.02	<0.02			
2F-1	-- <sup>5</sup>	0.02	<0.02			
2F-2	25	0.02	3.4			
2F-3	30	0.02	9.6			
2F-4	17	0.02	8.4			
2F-5	23	0.02	0.73			
2F-6	1.6	0.02	0.6			
2F-7	-- <sup>5</sup>	0.02	<0.02			
2G-1	-- <sup>5</sup>	0.02	<0.02			
2G-2	19	0.02	0.5			
2G-3	19	0.02	2.0			
2G-3(Dup) <sup>4</sup>	19	0.02	3.8			
2G-4	17	0.02	7.7			
2G-5	32	0.02	7.0			
2G-6	-- <sup>5</sup>	0.02	<0.02			
2H-1	-- <sup>5</sup>	0.02	<0.02			
2H-2	36	0.02	7.9			
2H-3	20	0.02	13			
2H-4	25	0.02	9.9			

TABLE 2-1 (continued)  
VERIFICATION SOIL SAMPLING RESULTS  
FOR AREA 2

Sample Description	September 3-4, 1987			October 16, 1987		
	Percent Moisture	PCBs <sup>1</sup> , D.L. <sup>2</sup>	mg/kg Conc <sup>3</sup>	Percent Moisture	PCBs <sup>1</sup> , D.L. <sup>2</sup>	mg/kg Conc <sup>3</sup>
2H-5	6.1	0.02	0.66			
2H-6	-- <sup>5</sup>	0.02	0.12			
2H-6 (Dup)	-- <sup>5</sup>	0.02	0.13			
BL-1	-- <sup>5</sup>	0.02	0.17			
BL-2	0.15	0.02	0.5			

**Notes:**

1. Only PCBs detected were Aroclor 1260.
2. D.L. is the detection limit
3. Conc. is the reported concentration of the PCB by the laboratory analysis.
4. Samples were taken by Department of Health Services in first round of sampling, split in DHS laboratory. One-half analyzed by DHS; other half sent to Central Coast Analytical Services for identical analysis.
5. Wet weight of the sample; other samples were air dried per Department of Health Services instructions.
6. Samples were taken by Department of Health Services in second round of sampling, split in DHS laboratory. One-half analyzed by DHS; other half sent to Central Coast Analytical Services for identical analysis.
7. ND = Not detected

TABLE 2-2

HUNTERS POINT ANNEX  
TREASURE ISLAND NAVAL STATION

VERIFICATION SOIL SAMPLING RESULTS  
SAMPLE COMPARISONS  
SEPTEMBER 3-4, 1987

Sample Description	Percent Moisture	DHS Lab		Central Coast Lab	
		PCBs <sup>1</sup> , D.L. <sup>2</sup>	mg/kg <sub>3</sub> Conc <sup>3</sup>	PCBs <sup>1</sup> , D.L. <sup>2</sup>	mg/kg <sub>3</sub> Conc <sup>3</sup>
2A-1	6.0	-- <sup>4</sup>	ND <sup>5</sup>	0.2	ND
2C-2	30	-- <sup>4</sup>	4	0.02	25
2C-5	<0.1	-- <sup>4</sup>	140	0.02	51
2C-6	4.7	-- <sup>4</sup>	ND	0.02	0.65
2E-1	5.8	-- <sup>4</sup>	ND	0.2	ND
2E-1 (Dup)	5.8	-- <sup>4</sup>	--	0.2	ND
2E-4	22	-- <sup>4</sup>	40	0.02	24
2E-4 (Dup)	22	-- <sup>4</sup>	--	0.02	25
2G-4	17	-- <sup>4</sup>	7.3	0.02	7.7
Instr. Blank	--	--	--	0.02	ND

## Notes:

1. Only PCBs detected were Aroclor 1260.
2. D.L. is the detection limit
3. Conc. is the reported concentration of the PCB by the laboratory analysis.
4. Detection limit not reported by DHS
5. ND = not detected

attained by the second round cleanup work. This resampling program was performed on October 16, 1987. At the request of the DHS, additional sampling locations within the excavation were established during the second round of verification sampling of Area 2. These locations are shown in Figure 2-2. Three additional samples were collected around grid location 2C-5, and are designated 2C-5a, 2C-5b and 2C-5c. Similarly, additional locations were established around 2E-3 (2E-3a, 2E-3b) and 2E-2 (2E-2a). All six of these additional sampling points were situated at locations midway between existing grid points.

DHS personnel were at the site during this period and took two samples (2C-5 and 2E-3) for splitting and for concurrent analysis by the DHS laboratory and Central Coast Analytical Services. As indicated in Table 2-1, the samples taken on October 16, 1987 and analyzed by Central Coast Analytical Services are reported to be below the cleanup criteria of 25 ppm.

#### Background and Blank Samples

As part of the quality assurance program, a soil background (BL-1) and blank (BL-2) were collected during the first round of Area 2 verification sampling. Soil sample BL-1 was collected from a location southwest of Area 2, approximately 40 ft. south-southwest of grid location 2A-1. The sample was obtained by pressing a California modified sample into undisturbed subsurface soils in a manner identical to that used to collect the September 3, 1987 samples around the periphery of the excavation.

Soil sample BL-2 consisted of an aliquot of soil previously prepared by Central Coast Analytical Services as a blank soil for the PCB Field Test kit analysis. Representative on-site soils were previously roasted to remove organics and then subjected to replicate analyses to verify the absence of PCBs. This soil sample serves as a true "blank" in that it consisted of a matrix typical of that found in actual verification samples but free of PCBs.

Trace levels of PCBs were detected in both soil samples BL-1 and BL-2. The occurrence of 0.17 mg/kg PCBs in BL-1 is possibly attributable to the presence of trace PCB levels in soils adjacent to the spill site. The presence of 0.5 mg/kg PCBs in BL-2 is possibly due to inadvertent contamination introduced into the soil blank during its repeated use in the Field Test kit analyses. Since the detection limit of the field kit is approximately 16 mg/kg, the trace concentration of 0.5 mg/kg found by EPA Method 8080 has no impact on the validity of the test kit data or the results of the verification sampling program.

It is apparent that both these occurrences are a result of factors not directly influencing the verification sample data. Compared to the PCB concentrations found in the actual verification samples, those found in samples BL-1 and BL-2 are not significant.

#### RECOMMENDATIONS

Based on the soil verification sampling results of September 3-4, 1987 and October 16, 1987, the area designated as Area 2 on Figure 2-1 meets the PCB interim cleanup criteria of 25 ppm. The area should be backfilled with approved material and surfaced with asphalt to match the existing area. A fence or other barrier should be constructed on the east side of area 2 to prevent equipment and personnel working in Area 3 from operating near the backfilled Area 2.

**References:**

1. PCB Verification Sampling Results, Interim Report, ERM-West, April 1987.
2. Addendum to the "PCB Verification Sampling Results", Attachment to a letter from the U.S. Navy to the Department of Health Services, June 25, 1987.
3. Verification of PCB Spill Cleanup by Sampling and Analysis, USEPA, August 1985.

**APPENDIX A**

**CHAIN OF CUSTODY RECORDS  
LABORATORY ANALYTICAL SUMMARIES**

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): D. Cufugno  
Date: 9-3-87  
Weather: Sunny, breezy, warm, dry

Job Location: San Fran CA  
Job No: 400-36  
No. of Samples Collected: 14 soils  
page 1 of 3

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

## Verification Samples : 5-day Turn around

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrn. Type	Preservative	Iced (X/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
✓ 2A-1 16"-2'0"	9:50A	D-6886	X	2" x 6"	1 brass tube	none	YES	shive sampler	PCBs
✓ 2G-1 18"-2'3"	10:15A		X	2" x 7"	1 steel tube				
✓ 2F-1 15"-2'0"	10:45A		X	2" x 7"	"				
✓ 2D-0 22"-2'8"	11:45A		X	2" x 6"	1 brass tube				
✓ 2B-6 16"-2'0"	12:55P		X	2" x 6"	"	✓	✓	✓	✓
✓ 2C-7 27"-3'1"	2:25P		X	2" x 6"	"	✓	✓	✓	✓

Comments: Analyze soil  
from bottom (recovered)  
depth of each hole  
Soil boxes ONLY! DO  
NOT INCLUDE PACKS  
CONTAINER ETC.  
Analyze 1 random sample  
in duplicate (1 of 14 total)

Custody Record  
Signature, Date/Time  
Relinquished: D. Cufugno 9-3-87 6:45P  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of  
Receiving Laboratory  
Central Coast  
Analytical Services (CCAS)  
141 Suburban Rd  
C-4  
San Luis Obispo CA  
93401  
(805) 543-2553

NOTE: Samples 2A-1, 2C-6 and 2E-1 were retained by the Dept.  
of Health Services. Splits of these samples will be sent to  
CCAS under separate cover... DC

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): Dr. C. C. C. C.  
Date: 9-3-87  
Weather: see p. 1

Job Location: San Fran CA  
Job No: 400-36  
No. of Samples Collected: 14 soils

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1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples: 5-day turn around

Sample ID #	Time	Sample Type		Volume	No. of Contnrs. Contnr. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
✓ 2D-6 16"-2'0"	2:35P		X	2" X 6"	1 steel tube	none	YES	drive sampler	PCBs
✓ 2E-7 18"-2'2"	2:50P		X		1 brass tube				
✓ 2F-7 1'0"-2'4"	3:10P		X		"				
✓ 2G-6 2'4"-2'10"	3:25P		X		"				
✓ 2H-6 1'4"-1'10"	3:50P		X		1 steel tube				
✓ 2I-6 1'8"-2'2"	4:10P		X		"				

Comments: \_\_\_\_\_  
see summary section  
pg 1

Custody Record  
Signature, Date/Time  
Relinquished: Dr. C. C. C. C. 9-3-87 6:45P  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: Michelle M. Miller 9-4-87 0900

Name and Address of Receiving Laboratory  
Central Coast  
Analytical Services  
San Luis Obispo CA

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): D. Cutugno  
Date: 9-3-87  
Weather: 52° 0-1

Job Location: San Francisco CA  
Job No: 400-36  
No. of Samples Collected: 14 soils

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1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

**Verification Samples: 5-day turn around**

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrr. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
✓ 2C-1 1'6"-2'0"	4:20P	17	6.8" X	2" X 6"	1 Grass tube	none	YES	drive sampler	PCBs
✓ BL-1 1'6"-2'0"	4:35P		6.8" X	"	"	"	"	"	"

Comments: \_\_\_\_\_  
see comment section,  
pg 1.  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Custody Record**  
**Signature, Date/Time**  
Relinquished: D. Cutugno 9-3-87 6:45P  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: Walter Miller  
9-4-87 0900

**Name and Address of Receiving Laboratory**  
\_\_\_\_\_  
Central Coast  
Analytical Services  
\_\_\_\_\_  
San Luis Obispo CA  
\_\_\_\_\_  
\_\_\_\_\_

Central  
Coast  
Analytical  
Services

Air, Water, & Hazardous Waste  
Sampling, Analysis, & Consultation  
State Certified Hazardous Waste,  
Chemistry, & Bacteriology Laboratory

141 Suburban Road, Suite C-4  
San Luis Obispo, Ca 93401  
(805) 543-2553 Fax (805) 543-2685

6483-D Calle Real  
Goleta, CA 93117  
(805) 964-7838

TELEFAX # 415 946-9968

COMPANY: ERM-West

ATTENTION: Dennis Miller

DATE: 11/17/89

FROM: Mary Hawlik

PAGE: 1 OF 12

SPECIAL RECEIVING INSTRUCTIONS:

method 3550 - we usually use  
this but use a #10 sieve (2mm x 2mm)  
instead of 1mm x 1mm (#10 is  
designated by Title 22, p. 1800.00.  
We do not air dry except for those  
samples split with Cal DOTS Lab  
because they wanted us to air dry.  
Our detection limits are such that a  
20g wet weight sample is used: mixed with  
Na<sub>2</sub>SO<sub>4</sub> then sonicated. Solvent is 1:1 hexane:  
acetone (B+3) chromatography grade.  
Sample is filtered and reduced in volume  
using Kuderna-Danish Concentrator.  
% moisture is determined under 7.2 of E 3550.

SENT BY: M/H

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Air, Water, & Hazardous Waste  
Sampling, Analysis, & Consultation  
State Certified Hazardous Waste,  
Chemistry, & Bacteriology Laboratory

141 Suburban Road, Suite C-4  
San Luis Obispo, CA. 93401  
(805) 543-2553 [TELEFAX #805-543-2685]

6483-D Calle Real  
Goleta, CA 93117  
(805) 964-7838

ATTENTION:

Dan C. Dennison

DATE:

9/17/87

FROM:

Mary

PAGE:

1 OF 2

## SPECIAL RECEIVING INSTRUCTIONS:

PCB ResultsD-6886 - D-6899

SENT BY: \_\_\_\_\_

c[&lt;1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of PUBLIC HEALTH

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553

Lab Number: As Listed  
Collected: 09/03/87  
Received: 09/04/87  
Tested: 09/07/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94598

Sample Description:

U.S. Navy Hunter's Point  
Project #400-36

## REPORT

LAB NUMBER

SAMPLE DESCRIPTION

TOTAL  
LEVEL FOUND-  
milligrams/kg  
(Wet Weight)

## POLYCHLORINATED BIPHENYLS

EPA METHOD NUMBER-----  
DETECTION LIMIT-----  
DATE TESTED/ANALYST-----

0000  
0.02  
9/7/87/ARP

D-6886	2H-1 @ 1'6"-2'0"	<0.02
D-6887	2G-1 @ 1'8"-2'3"	<0.02
D-6888	2F-1 @ 1'5"-2'0"	<0.02
D-6889	2D-0 @ 2'3"-2'8"	<0.02
D-6890	2B-6 @ 1'6"-2'0"	<0.02
D-6891	2C-7 @ 2'7"-3'1"	<0.02
D-6892	2D-6 @ 1'6"-2'0"	<0.02
D-6893	2E-7 @ 1'8"-2'2"	<0.02
D-6894	2F-7 @ 1'10"-2'4"	<0.02
D-6895	2G-6 @ 2'4"-2'10"	<0.02
D-6896	2H-6 @ 1'4"-1'10"	0.12
QD-6896	DUPLICATE	0.13
D-6897	2B-1 @ 1'8"-2'2"	<0.02
D-6898	2C-1 @ 1'6"-2'0"	0.04
D-6899	BL-1 @ 1'6"-2'0"	0.17

D6886ERM.WR1  
MH/ke

Respectfully submitted,  
CENTRAL COAST ANALYTICAL SERVICES  
*Mary Havlicek*  
Mary Havlicek, Ph.D., President



Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-7239  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#500,2A-1, %m=6.8;%s=20  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1260	0.2	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D7239PC.WR1  
MH/TK/ARP/ARP

Central  
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Analytical  
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Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-7240  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:

U.S. Navy Hunters Point  
HML#502, 2E-1, %m=5.8; %s=15  
PROJECT #400-36

s/be HML# 501

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1260	0.2	not found

Compounds listed as "not found" would have been reported if present at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D7240PC.WR1  
MH/TK/ARP/ARP

Central  
Coast  
Analytical  
Services

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Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QD-7240  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#502,2E-1, ~~Sm~~=5.8;~~Ss~~=15  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1260	0.2	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

QD7240PC.WR1  
MH/TK/ARP/ARP

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-7241  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Don Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#502,2C-6, %m=4.7;%s=19  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	0.65

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

  
Mary Havlicek, Ph.D.

D7241PC.WR1  
MH/TK/ARP/ARP

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler (s): D. Cufagno/R. Knapp  
Date: 9/4/87  
Weather: partly cloudy, warm, breezy, dry

Job Location: San Fran, CA  
Job No: 400-36  
No. of Samples Collected: 31 soils / 2 waters  
page 1 of 1

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples : 5-day turnaround

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contr. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab Comp. Grab						
6950 2A-2	2:13 P		X	~40z.	1 glass jar	none	YES	manual	PCBs
951 2A-3	2:17 P		X						
952 2A-4	2:22 P		X						
953 2B-2	2:20 P		X						
954 2B-3	2:18 P		X						
955 2B-4	2:15 P		X						

Comments:

31 ~~soils~~ soils : analyzed  
10% in duplicate  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## Custody Record

Signature, Date/Time

Relinquished: D. Cufagno 9/4/87 GPM  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of Receiving Laboratory

Central Coast  
Analytical Services  
141 Suburban Pk  
C-4  
San Luis Obispo CA  
93401  
(805) 543-2553

NOTE: Samples 2C-2, 2C-5, 2E-4 and 2G-4 were retained by the Dept. of Health Services; splits will be sent to CCAS under separate cover.

APPENDIX A  
CHAIN OF CUSTODY RECORDS  
LABORATORY ANALYTICAL SUMMARIES –  
VERIFICATION SAMPLES  
PAGE 1 OF 6

POLYCHLORINATED BIPHENYL VERIFICATION  
SAMPLING RESULTS  
AREA 2

THE ABOVE IDENTIFIED PAGE IS NOT AVAILABLE.

EXTENSIVE RESEARCH WAS PERFORMED BY  
NAVFAC SOUTHWEST TO LOCATE THIS PAGE.  
THIS PAGE HAS BEEN INSERTED AS A  
PLACEHOLDER AND WILL BE REPLACED SHOULD  
THE MISSING ITEM BE LOCATED.

FOR ADDITIONAL INFORMATION,  
PLEASE CONTACT:

**DIANE C. SILVA, RECORDS MANAGER  
NAVAL FACILITIES ENGINEERING COMMAND  
SOUTHWEST  
1220 PACIFIC HIGHWAY  
SAN DIEGO, CA 92132**

**TELEPHONE: (619) 532-3676  
E-MAIL: [diane.silva@navy.mil](mailto:diane.silva@navy.mil)**

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): Cutugno/Krapp  
Date: 9/4/87  
Weather: sea p. 1

Job Location: San Fran CA  
Job No: 400-36  
No. of Samples Collected: 3.1 soils/2 waters

page 2 of 6

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples: 5-day turnaround

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrr. Type	Preservative	Iced (X/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
956 2B-5	2:28P		X	~4 oz.	1 glass jar	none	YES	manual	PCBs
957 2C-3	2:24P		X						
958 2C-4	2:24P		X						
959 2D-1	2:36P		X						
960 2D-2	2:39P		X						
961 2D-3	2:35P		X						

Comments: \_\_\_\_\_

sea p. 1  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Custody Record  
Signature, Date/Time  
Relinquished: D. Cutugno 9/4/87 6PM  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of Receiving Laboratory

Central Coast  
Analytical  
San Luis Obispo CA  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): Cutugno/Enapp  
Date: 9/4/87  
Weather: see p. 1

Job Location: San Fran CA  
Job No: 400-36  
No. of Samples Collected: 31 with 20.75

page 3 of 6

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples: 5-day turnaround

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrr. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
26961 2D-4	2:43 P		X	~4 oz.	1 glass jar	none	YES	manual	ACBr
6963 2D-5	2:12 P		X						
6964 2E-2	2:48 P		X						
6965 2E-3	2:49 P		X						
6966 2E-5	2:56 P		X						
6967 2E-6	3:10 P		X						

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
see p. 1  
\_\_\_\_\_  
\_\_\_\_\_  
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\_\_\_\_\_  
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\_\_\_\_\_

Custody Record  
Signature, Date/Time  
Relinquished: D. Cutugno 9/4/87 GPM  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of Receiving Laboratory  
\_\_\_\_\_  
Central Coast  
Analytical  
\_\_\_\_\_  
San Luis Obispo  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): C. H. Young / Knapp  
Date: 7/4/87  
Weather: see p. 1

Job Location: San Fran CA  
Job No: 400-36  
No. of Samples Collected: 31 soils / 2 waters

page 4 of 6

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples: 5-day turnaround

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrn. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
D6468 2F-2	2:50 P		X	~4 oz	1 glass jar	none	YES	manual	PCBs
6969 2F-3	2:53 P		X						
6970 2F-4	3:31 P		X						
971 2F-5	3:20 P		X						
972 2F-6	3:35 P		X						
973 2G-2	3:01 P		X						

Comments: \_\_\_\_\_  
\_\_\_\_\_  
see p. 1  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Custody Record  
Signature, Date/Time  
Relinquished: H. Cantuero 9/4/87 GPM  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of Receiving Laboratory  
\_\_\_\_\_  
Central Coast  
\_\_\_\_\_  
Analytical Services  
\_\_\_\_\_  
San Luis Obispo CA  
\_\_\_\_\_  
\_\_\_\_\_

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): C. Alvarez / Knapp  
Date: 9/14/87  
Weather: see p. 1

Job Location: San Fran CA  
Job No: 400-36  
No. of Samples Collected: 31 soils / 2 waters  
page 5 of 6

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

*Verification Samples: 5-day turnaround*

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrr. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
06974 2G-3	2:55P		X	~402.	1 glass jar	none	YES	manual	PCBs
6975 2G-5	3:23P		X						
6976 2H-2	3:02P		X						
6977 2H-3	3:14P		X						
6978 2H-4	3:17P		X						
6979 2H-5	3:43P		X						
6980 BL-2	5:00P		X	~202.	1 glass jar	none	YES	manual	PCBs

Comments: \_\_\_\_\_  
\_\_\_\_\_  
see p. 1  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Custody Record**  
**Signature, Date/Time**  
Relinquished: D. Cantueros 9/14/87 6PM  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

**Name and Address of Receiving Laboratory**  
\_\_\_\_\_  
Central Coast  
Analytical  
\_\_\_\_\_  
San Luis Obispo CA  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

# CHAIN OF CUSTODY AND SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Nav.  
Sampler (s): C. L. Turner  
Date: 9/4/87  
Weather: see p. 1

Job Location: San Francisco  
Job No: 400-36  
No. of Samples Collected: 31 soils/2 waters

page 6 of 6

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

**5-day turnaround**

Sample ID #	Time	Sample Type		Soil		Volume	No. of Contrs. Contrn. Type	Preservative	Iced (Y/N)	Sampling Method	Analyses
		Water	Grab	Comp.	Grab						
06981 #1	9:45A		X			12	1 amber glass bottle	none	YES	manual	see comment section
06982 #2	3:20P		X			12	"	"	"	"	"

Comments: \_\_\_\_\_

PCB Analysis:  
#1: supernatant  
only: DO NOT stir  
SEDIMENT  
#2: Total contents

Custody Record  
Signature, Date/Time  
Relinquished: D. C. Turner 9/4/87 GPM  
Received: [Signature]  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of Receiving Laboratory  
\_\_\_\_\_  
Central Coast  
Analytical  
P.O. Box 1010  
San Luis Obispo CA  
\_\_\_\_\_  
\_\_\_\_\_

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553

Lab Number: As Listed  
Collected: 09/04/87  
Received: 09/05/87  
Tested: As Listed  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94523

Sample Description:

Hunter's Point, U.S. Navy  
Project #400-36  
Samples As Listed

REPORT

LAB NUMBER	SAMPLE DESCRIPTION	PERCENT MOISTURE	POLYCHLORINATED BIPHENYLS AROCOR 1260
EPA METHOD NUMBER-----		Percent	mg/kg (Wet Weight)
DETECTION LIMIT-----		160.3	8080
DATE TESTED/ANALYST-----		0.005	0.02
		9/11/87/LD	9/08-15/87/ARP
D-6950	2A-2	3.2	5.0
D-6951	2A-3	7.1	2.2
D-6952	2A-4	2.7	0.61
D-6953	2B-2	16.	16.
D-6954	2B-3	7.2	0.44
QD-6954	DUPLICATE	7.1	---
D-6955	2B-4	5.1	1.0
D-6956	2B-5	3.8	0.60
D-6957	2C-3	12.	3.2

D6950ERM.WR1  
MH/ke

Respectfully submitted,  
CENTRAL COAST ANALYTICAL SERVICES  
*Mary Havlicek*  
Mary Havlicek, Ph.D., President

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553

Lab Number: As Listed  
Collected: 09/04/87  
Received: 09/05/87  
Tested: As Listed  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94523

Sample Description:  
  
Hunter's Point, U.S. Navy  
Project #400-36  
Samples As Listed

# REPORT

LAB NUMBER	SAMPLE DESCRIPTION	LEVEL FOUND -	
		PERCENT MOISTURE	POLYCHLORINATED BIPHENYLS AROCOR 1260
EPA METHOD NUMBER-----		Percent	mg/kg (Wet Weight)
DETECTION LIMIT-----		160.3	8080
DATE TESTED/ANALYST-----		0.005	0.02
		9/11/87/LD	9/08-15/87/ARP
D-6958	2C-4	25.	15.
D-6959	2D-1	30.	3.5
D-6960	2D-2	41.	21.
D-6961	2D-3	31.	4.3
D-6962	2D-4	21.	2.2
D-6963	2D-5	1.2	2.2
D-6964	2E-2	41.	36.
D-6965	2E-3	25.	59.
D-6966	2E-5	20.	4.6
QD-6966	DUPLICATE	20.	4.2
D-6967	2E-6	17.	0.60
D-6968	2F-2	25.	3.4
D-6969	2F-3	30.	9.6

D6958ERM.WR1  
MH/ke

Respectfully submitted,  
CENTRAL COAST ANALYTICAL SERVICES  
*Mary Havlicek*  
Mary Havlicek, Ph.D., President

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553

Lab Number: As Listed  
Collected: 09/04/87  
Received: 09/05/87  
Tested: As Listed  
Collected by: Richard Knapp

ERM-WEST  
1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94523

Sample Description:  
  
Hunter's Point, U.S. Navy  
Project #400-36  
Samples As Listed

# REPORT

LAB NUMBER	SAMPLE DESCRIPTION	LEVEL FOUND -	
		PERCENT MOISTURE	POLYCHLORINATED BIPHENYLS AROCOR 1260
EPA METHOD NUMBER-----		Percent	mg/kg (Wet Weight)
DETECTION LIMIT-----		160.3	8080
DATE TESTED/ANALYST-----		0.005	0.02
		9/11/87/LD	9/11/87/ARP
D-6970	2F-4	17.	8.4
D-6971	2F-5	23.	0.73
D-6972	2F-6	1.6	0.6
D-6973	2G-2	19.	0.50
D-6974	2G-3	19.	2.0
QD-6974	DUPLICATE	19.	3.8
D-6975	2G-5	32.	7.0
D-6976	2H-2	36.	7.9
D-6977	2H-3	20.	13.0
D-6978	2H-4	25.	9.9
D-6979	2H-5	6.1	0.66
D-6980	BL-2	0.15	0.50

D6970ERM.WR1  
MH/ke

Respectfully submitted,  
CENTRAL COAST ANALYTICAL SERVICES  
*Mary Havicek*  
Mary Havicek, Ph.D., President

HAZARDOUS MATERIALS SAMPLE ANALYSIS REQUEST		All applicable items must be completed		1. HML No. To: <u>DS09-D12</u>		2. Page Z of 2							
3. Collector <u>Chien Kao</u>		4. Phone <u>415 540-3052</u>		5. Priority <input checked="" type="checkbox"/>		6. Authorized by _____							
6. Date Sampled <u>9/4/87</u>		7. Time Sampled _____ Hours		8. Codes (fill in all applicable codes)									
9. Activity <input type="checkbox"/> Ent <input type="checkbox"/> Surv <input checked="" type="checkbox"/> Site Mit <input type="checkbox"/> Permitting <input type="checkbox"/> Alt Tech <input type="checkbox"/> Other				a. STC <u>001021</u>									
10. SAMPLING LOCATION				b. Region <u>4</u>									
a. Site <u>Hunters Point Naval Shipyard</u>		a. EPA ID No.		c. TPC _____									
c. Address <u>S.F.</u>				d. INDEX <u>6530</u>									
Number _____ Street _____ City _____ Zip _____				e. PCA <u>11025</u>									
				f. SITE <u>2000500</u>									
				g. County <u>075</u>									
11. SAMPLES													
a. ID		b. Collector's No.		c. HML No.		d. Type		e. Type		f. Size		g. Field Information	
<u>AD242C-5</u>		<u>DS09</u>		<u>DS10</u>		<u>Soil</u>		<u>G</u>		<u>250</u>			
<u>B3 2E4</u>		<u>DS10</u>		<u>DS11</u>		<u>"</u>		<u>G</u>		<u>"</u>			
<u>C. 44 2G-4</u>		<u>DS11</u>		<u>DS12</u>		<u>"</u>		<u>G</u>		<u>"</u>			
<u>D. 45 2C-2</u>		<u>DS12</u>				<u>"</u>		<u>G</u>		<u>"</u>			
<u>E</u>													
<u>F</u>													
<u>H</u>													
12. ANALYSIS REQUESTED								f. <input checked="" type="checkbox"/> PCB		k. <input type="checkbox"/> Ext. Org (Screening)			
a. <input type="checkbox"/> pH								g. <input type="checkbox"/> VOA		l. <input type="checkbox"/> Chlorinated Pesticides			
b. <input type="checkbox"/> Metal Scan								h. <input type="checkbox"/> PAH		m. <input type="checkbox"/> Organo-P Pesticides			
c. <input type="checkbox"/> Metals (Spec)								i. <input type="checkbox"/> Phenols		n. <input type="checkbox"/>			
d. <input type="checkbox"/> W.E.T.								j. <input type="checkbox"/> Carbamates		o. <input type="checkbox"/>			
13. CHAIN OF CUSTODY													
1. <u>Chien Kao</u>		<u>Chien Ping Kao / AWM</u>		<u>9/4/87 - 9/10/87</u>									
Signature		Name/Title		Inclusive Dates									
2. <u>J. Thom Coons</u>		<u>J. Thom Coons / Chemist</u>		<u>9/14/87 - 9/14/87</u>									
Signature		Name/Title		Inclusive Dates									
3. <u>Bruce P. Hershman</u>		<u>VERNE FARR / ABBSIT</u>		<u>9/4/87 - 9/15/87</u>									
Signature		Name/Title		Inclusive Dates									
4. <u>Chien Kao</u>		<u>Chien Ping Kao / AWM</u>		<u>9/15/87 - 9/16/87</u>									
Signature		Name/Title		Inclusive Dates									
5. <u>Shellie Noller</u>		<u>Shellie Noller / Sample Control</u>		<u>9/17/87</u>									
Signature		Name/Title		Inclusive Dates									
14. SPECIAL REMARKS <u>Security seal intact upon receipt.</u>													
15. RECEIVED BY <u>[Signature]</u>								a. Title <u>AWM</u>		b. Date <u>9-8-87</u>			
16. SAMPLE ALLOCATION a. <input type="checkbox"/> HML-Berkeley b. <input type="checkbox"/> HML-SC c. <input type="checkbox"/> AIHL d. <input type="checkbox"/> Contract b. Date													
17. ANALYSIS REQUESTED <u>SCB</u>													

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-7242  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

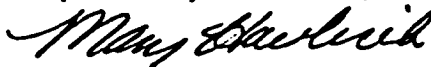
ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#509,2C-5, %m<0.1;%s=17  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	51.

Compounds listed as "not found", would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,



Mary Havlicek, Ph.D.

D7242PC.WR1  
MH/GH/ARP/ARP

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141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-7243  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#510, 2E-4, 7m=22; 7s=40  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	24.

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D7243PC.WR1  
MH/TK/ARP/ARP

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Analytical Services  
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San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QD-7243  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#510,2E-4, %m=22;%s=40  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	25.

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,



Mary Havlicek, Ph.D.

QD-7243PC.WR1  
MH/TK/ARP/ARP

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EPA METHOD 608/8080 - PCB'S

Lob Number: D-7244  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#511,2G-4, %m=17;%s=48  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	7.7

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D7244PC.WR1  
MH/TK/ARP/ARP

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San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-7245  
Collected: 9/04/78  
Received: 9/17/87  
Tested: 9/21/87  
Collected by: Dan Catugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
U.S Navy Hunters Point  
HML#512,2C-2, %m=30;%s=38  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	25.

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 9/18/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D7245PC.WR1  
MH/TK/ARP/ARP

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(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: B-09217  
Collected:  
Received:  
Tested: 9/21/87  
Collected by:

CCAS

SAMPLE DESCRIPTION:  
Instrument Blank

PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.02	not found
PCB 1221	0.02	not found
PCB 1232	0.02	not found
PCB 1242	0.02	not found
PCB 1248	0.02	not found
PCB 1254	0.02	not found
PCB 1260	0.02	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits.

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

## CHAIN OF CUSTODY

## SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): D. Cutugno  
Date: 10-16-87  
Weather: sunny, warm, dry

Job Location: Hunters Pt., San Fran.  
Job No: 400-36  
No. of Samples Collected: 11\*

page 1 of 2

1777 Botalho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples: 5-day turnaround

Sample ID #	Time	Sample Type		Volume	No. of Contnrs. Contnr. Type	Preservative	Iced (X/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
2C-5a	12:31P		X	~602.	1 glass jar	none	YES	manual	see comment section
2C-5b	12:32P		X						
2C-5c	12:34P		X						
2E-4	12:36P		X						
2D-2	12:37P		X						
2E-3b	12:39		X						

## Comments:

Analyze for PCBs;  
if possible, AAR DAY  
prior to screening  
and homogenizing  
Analyze 2E-2 in  
TRIPPLICATE

## Custody Record

Signature, Date/Time

Relinquished: D. Cutugno 10-16-87  
Received: 2:45P  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

## Name and Address of Receiving Laboratory

Central Coast  
Analytical Services  
141 Suburban Rd  
C-4  
San Luis Obispo CA  
93401  
(805) 543-2553

\* NOTE: Samples 2E-3 and 2C-5 were retained by CA.  
DHS and will be sent to CCAS under separate  
cover. AS

## CHAIN OF CUSTODY

## SAMPLE IDENTIFICATION RECORD

ERM-West  
Environmental  
Resources  
Management

Client: Navy  
Sampler(s): D. Cutugno  
Date: 10-16-87  
Weather: Sunny, warm, dry

Job Location: Hunters Pt., San Fran.  
Job No: 400-36  
No. of Samples Collected: 11 (see p. 1)

page 2 of 2

1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596  
(415) 946-0455

Verification Samples: 5-day turnaround

Sample ID #	Time	Sample Type		Volume	No. of Contrs. Contrr. Type	Preservative	Iced (X/N)	Sampling Method	Analyses
		Water Comp.	Soil Grab						
2E-3a	12:42P		X	~602.	1 glass jar	none	YES	manual	see comment section pg 1.
2E-2a	12:45P		X	↓	↓	↓	↓	↓	↓
2E-2	1:05P		X	↓	↓	↓	↓	↓	↓

Comments:

\_\_\_\_\_  
\_\_\_\_\_  
see pg. 1  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Custody Record

Signature, Date/Time

Relinquished: D. Cutugno 10-16-87  
Received: 2:45P  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_  
Relinquished: \_\_\_\_\_  
Received: \_\_\_\_\_

Name and Address of Receiving Laboratory

Central Coast  
Analytical Svs.  
San Luis Obispo CA  
\_\_\_\_\_  
\_\_\_\_\_

OCT 26 '87 15:12 C C R S

P02

c[&lt;1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of PUBLIC HEALTH

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553

Lab Number: As Listed  
Collected: 10/16/87  
Received: 10/17/87  
Tested: As Listed  
Collected by: D. Catugno

ERM-WEST  
1777 Botelho Drive  
Suite 260  
Walnut Creek, CA 94596

Sample Description:  
  
US Navy, Hunter's Point  
Project #400-38, S.F.

## REPORT

## LAB NUMBER

## SAMPLE DESCRIPTION

## LEVEL FOUND-

EPA METHOD NUMBER-----  
DETECTION LIMIT-----  
DATE TESTED/ANALYST-----

PERCENT MOISTURE %	PERCENT SOLID %
160.3	160.3
0.005	0.005
10/21/87/RDM	10/21/87/RDM

D-8233	Sample #2C-5A	15.	85.
D-8234	Sample #2C-5B	21.	79.
D-8235	Sample #2C-5C	19.	81.
D-8236	Sample #2E-4	19.	81.
D-8237	Sample #2D-2	24.	76.
D-8238	Sample #2E-3B	16.	84.
D-8239	Sample #2E-3A	20.	80.
D-8240	Sample #2E-2A	5.	95.
D-8241	Sample #2E-2	18.	82.

D8233ERM.WR1  
MH/ke

Respectfully submitted,  
CENTRAL COAST ANALYTICAL SERVICES  
*Mary Havlicek*  
Mary Havlicek, Ph.D., President

OCT 26 '87 15:21 C C R S

P15

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

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Coast  
Analytical  
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Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 658/8585 - PCB'S

Lab Number: QO-8241  
Collected: 10/16/87  
Received: 10/17/87  
Tested: 10/23/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Batelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2C-5A  
PROJECT #488-38

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	2.9

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

OCT 26 '87 15:13 C C A S

P03

<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICES

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Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8233  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/22/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2C-5A  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	2.4

Compounds listed as "not found" would have been reported if present at or above the listed detection limits. Sample was extracted 10/20/87 .

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QT-8241  
Collected: 10/16/87  
Received: 10/17/87  
Tested: 10/22/87  
Collected by: Don Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2C-5A  
PROJECT #400-38

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	2.7

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87.

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

OCT 26 '87 15:14 C C R S

P04

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

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Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB's

Lab Number: D-8234  
Collected: 10/16/87  
Received: 10/17/87  
Tested: 10/22/87  
Collected by: Dan Cutugno

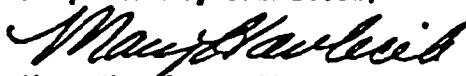
ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2C-58  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	11.

Compounds listed as "not found" would have been reported if present at or above the listed detection limits. Sample was extracted 10/20/87.

Respectfully submitted,

  
Mary Havicek, Ph.D.

OCT 26 '87 15:14 C C R S

P05

c[<1[18a AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

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(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8235  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/22/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 200  
Walnut Creek, CA 94598

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2C-5C  
PROJECT #400-38

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	1.5

Compounds listed as "not found" would have been reported if present at or above the listed detection limits. Sample was extracted 10/20/87.

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

OCT 26 '87 15:15 C C R S

P06

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

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Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8236  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/22/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2E-4  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	8.9

Compounds listed as "not found" would have been reported if present at or above the listed detection limits. Sample was extracted 10/20/87 .

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D8236PC.WR1  
MH/JK/ARP/ARP

OCT 26 '87 15:16 C C R S

P07

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QS-8236  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/23/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94598

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point, 2E-4,  
Spiked at 4.4 ppm  
PROJECT #400-38

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	14.*

\*Recovery of spike = 116%

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

QS8236PC.WR1  
MH/GH/ARP/ARP

OCT 26 '87 15:16 C C R S

P08

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 600/8-80-01 - PCB'S

Lab Number: D-8237  
Collected: 10/16/87  
Received: 10/17/87  
Tested: 10/22/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
20-2  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	16.

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87.

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D8237PC.WR1  
MH/JK/ARP/ARP

OCT 26 '87 15:17 C C R S

P09

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

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Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8238  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/21/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2E-3B  
PROJECT #400-38

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	0.79

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D8238PC.WR1  
MH/GH/ARP/ARP

OCT 26 '87 15:18 C C R S

P10

c[<1]10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERV

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Analytical  
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Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8239  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/21/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2E-3A  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	15.

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D8239PC.WR1  
MH/GH/ARP/ARP

OCT 26 '87 15:18 C C R S

P11

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERV

Central  
Coast  
Analytical  
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Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8240  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/21/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94598

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2E-2A  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	2.2

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

D8240PC.WR1  
MH/JJK/ARP/ARP

OCT 26 '87 15:19 C C R S

P12

c[<1[19m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERV

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Analytical  
Services

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Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/808# - PCB'S

Lab Number: D-8241  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/21/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2E-2  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	3.0

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

OCT 26 '87 15:20 C C R S

P13

c[<1[15m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVI

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QD-8241  
Collected: 10/16/87  
Received: 10/16/87  
Tested: 10/21/87  
Collected by: Dan Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

SAMPLE DESCRIPTION:  
Erm West; Navy, Hunter's Point  
2E-2  
PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	3.0

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

OCT 26 '87 15:22 C C R S

P16

c[<1[15m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT OF HEALTH SERV

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 600/8080 - PCB'S

Lab Number: QH10207  
Collected:  
Received:  
Tested: 10/21/87  
Collected by:

CCAS

SAMPLE DESCRIPTION:  
Sonic Horn Extraction Blank

PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

QH10207PC.WR1  
MH/JJK/ARP/ARP

OCT 26 '87 15:23 C C R S

P17

c[<1[15 AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVI

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Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: B10217  
Collected:  
Received:  
Tested: 10/21/87  
Collected by:

CCAS

SAMPLE DESCRIPTION:  
Instrument Blank

PROJECT #400-36

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

B10217PC.WR1  
JH/JJK/ARP/ARP

OCT 26 '87 15:23 C C R S

P18

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT OF HEALTH SERVICE

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Analytical Services  
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San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: B-10227  
Collected: na  
Received: na  
Tested: 10/22/87  
Collected by: na

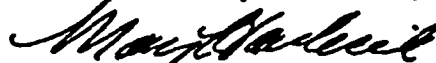
CCAS

SAMPLE DESCRIPTION:  
Instrument Blank

Compound Analyzed	Detection Limit micrograms/L	Concentration micrograms/L
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1260	0.2	not found

Compounds listed as "not found" would have been reported if present at or above the listed detection limits.

Respectfully submitted,



Mary Havlicek, Ph.D.

OCT 26 '87 15:24 C C R S

P19

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVI

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QL-1005  
Collected: 10/05/87  
Received: 10/05/87  
Tested: 10/22/87  
Collected by: na

CCAS

SAMPLE DESCRIPTION:  
Liquid-Liquid Extraction Blank

Compound Analyzed	Detection Limit micrograms/L	Concentration micrograms/L
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1260	0.2	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/05/87 .

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

QL1005pc.wr1  
MH/GH/ARP/ARP

OCT 26 '87 15:25 C C R S

P20

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SER

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: B-10237  
Collected:  
Received:  
Tested: 10/23/87  
Collected by:

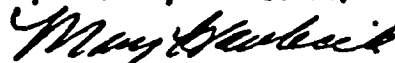
CCAS

SAMPLE DESCRIPTION:  
Instrument Blank

Compound Analyzed	Detection Limit micrograms/Kg	Concentration micrograms/Kg
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1268	0.2	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits.

Respectfully submitted,



Mary Havlicek, Ph.D.

OCT 26 '87 15:26 C C R S

P21

c[<1[10m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: QL-1002  
Collected:  
Received:  
Tested: 10/23/87  
Collected by:

CCAS

SAMPLE DESCRIPTION:  
Liq:Liq Extraction Blank

Compound Analyzed	Detection Limit micrograms/Kg	Concentration micrograms/Kg
PCB 1016	0.2	not found
PCB 1221	0.2	not found
PCB 1232	0.2	not found
PCB 1242	0.2	not found
PCB 1248	0.2	not found
PCB 1254	0.2	not found
PCB 1260	0.2	not found

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/20/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

QL1002PC.WR1  
MH/GH/ARP/ARP

HAZARDOUS MATERIALS  
SAMPLE ANALYSIS REQUESTAll applicable items  
must be completed

1. HML No.

To

D723-D724

2. Page

of

3. Collector

Chein Kao

4. Phone 415 540 3052

5. Priority ☐

a. Authorized by

6. Date Sampled

10/16/87

7. Time Sampled 12.30 Hours

8. Codes (fill in all applicable codes)

9. Activity

☐ Ent☐ Surv☒ Site Mit☐ Permitting☐ All Tech☐ Other

a. STC

b. Region

c. TPC

d. INDEX

e. PCA

f. SITE

g. County

0	0	1	0	2	1
4					
6	5	3	0		
1	1	0	2	5	
2	0	0	0	5	0
0	7	5			

10. SAMPLING LOCATION

2. EPA ID No.

b. Site

Hunters Point Naval Shipyard

c. Address

S.F.

Number

Street

City

Zip

11. SAMPLES

a. ID	b. Collector's No.	c. HML No.	d. Type	e. Type	f. Size	g. Field Information
A.	2C-5	D723	SOI	G	250	resample the same locat
B.	2E-3	D724	SOI	G	250	" " "
C.						
D.						
E.						
F.						
G.						
H.						

ANALYSIS REQUESTED

a. ☐ pHb. ☐ Metal Scanc. ☐ Metals (Spec)d. ☐ W.E.T.f. ☒ PCBg. ☐ VOAh. ☐ PAHi. ☐ Phenolsj. ☐ Carba-matesk. ☐ Ext. Org (Screening)l. ☐ Chlorinated Pesticidesm. ☐ Organo-P Pesticidesn. ☐o. ☐

13. CHAIN OF CUSTODY

a. Chein Kao  
Signature

b. Howard S. Okamoto  
Signature

c. Chein Kao  
Signature

d. Shella Miller  
Signature

Chein Ping Kao / AWME

10/16/87 - 10/16/88

Inclusive Dates

VERDE EARL / AWME

10/16/87 - 10/23/88

Inclusive Dates

Chein Ping Kao / AWME

10/23/87 - 1/1/88

Inclusive Dates

Shella Miller / Sample Control - CCAS

10/26/87 - 1/1/88

Inclusive Dates

14. SPECIAL REMARKS

please contact Howard Okamoto for special instructions

15. RECEIVED BY

Howard S. Okamoto

a. Title

PHC III

b. Date

10/16/87

16. SAMPLE ALLOCATION

a. ☐ HML-Berkeleyb. ☐ HML-SCc. ☐ AIHLd. ☐ Contract

b. Date

17. ANALYSIS REQUESTED

PCBs

Air dried #10 mesh fraction - splits to be given to C.P. Kao

c[&lt;1[18m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

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Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 608/8080 - PCB'S

Lab Number: D-8553  
Collected: 10/16/87  
Received: 10/28/87  
Tested: 11/05/87  
Collected by: D. Cutugno

ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94596

## SAMPLE DESCRIPTION:

2E-3, Hunter's Point, Soil

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.05	not found
PCB 1221	0.05	not found
PCB 1232	0.05	not found
PCB 1242	0.05	not found
PCB 1248	0.05	not found
PCB 1254	0.05	not found
PCB 1260	0.05	7.9

Compounds listed as "not found" would have been reported if present  
at or above the listed detection limits. Sample was extracted 10/28/87

Respectfully submitted,

*Mary Havlicek*  
Mary Havlicek, Ph.D.

c[&lt;1[16m AIR, WATER and HAZARDOUS WASTE LABORATORY CERTIFIED by CALIFORNIA DEPT of HEALTH SERVICE

Central  
Coast  
Analytical  
Services

Central Coast  
Analytical Services  
141 Suburban Road , Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553  
EPA METHOD 808/8080 - PCB'S

Lab Number: D-8552  
Collected: 10/16/87  
Received: 10/26/87  
Tested: 11/05/87  
Collected by: Dan Cutugno

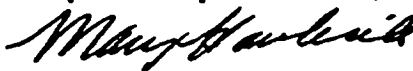
ERM-WEST  
1777 Botelho Dr., Suite 260  
Walnut Creek, CA 94598

SAMPLE DESCRIPTION:  
2c-5, Hunter's Point, Soil

Compound Analyzed	Detection Limit milligrams/Kg	Concentration milligrams/Kg
PCB 1016	0.1	not found
PCB 1221	0.1	not found
PCB 1232	0.1	not found
PCB 1242	0.1	not found
PCB 1248	0.1	not found
PCB 1254	0.1	not found
PCB 1260	0.1	1.3

Compounds listed as "not found" would have been reported if present at or above the listed detection limits. Sample was extracted 10/28/87

Respectfully submitted,

  
Mary Havlicek, Ph.D.

## **APPENDIX B**

### **LABORATORY QA/QC PROCEDURES**

Central  
Coast  
Analytical  
Services

**CENTRAL COAST  
ANALYTICAL SERVICES**  
Air, Water & Hazardous Waste Analysis  
141 Suburban Road, Suite C-4  
San Luis Obispo, California 93401  
(805) 543-2553

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NOV 13 1987

ERM-WEST  
WALNUT CREEK, CA

August 1, 1986

**QUALITY ASSURANCE/QUALITY CONTROL PROCEDURE**

1. Chain of Custody samples are stored in locked refrigerators.
2. The assignment of unique laboratory numbers to each sample as it is logged in has been extended to include the assignment of unique log numbers to subsamples on a container-by-container basis.
3. A central standard logbook is maintained for all standards. Information such as suppliers, lot numbers, weight/volume of standards used, date prepared and the name of the analyst preparing the standard are all part of the records kept therein.
4. The laboratory runs calibration standards at a minimum of three concentrations. We bracket sample data with standards.
5. We confirm all positive gas chromatographic results by either a second column or by GC/MS.
6. Blanks, duplicates, and spikes are analyzed once per batch, once per matrix type or once per 20 samples, whichever is more frequent. We have adopted an "all with the final report" filing system.
7. Records of the analysis of blank samples are recorded in individual laboratory notebooks, referred to on laboratory worksheets and documented on instrument use record books.
8. Acceptance limits for quality control samples currently used by our laboratory are summarized in Table I.
9. All analytical and quality control results are reviewed and approved by a supervisor. Data of an unusual nature are brought to the attention of one of our three Ph.D. chemists for final review.
10. Instructions for corrective action in the event of an out-of-control method are as follows: Stop analyses. Conduct investigation. Check mathematics. Check dilutions for systematic error. Check syringes, automatic pipettes, ect. for possible malfunction. Check dates on standards. Remake standards. If instrument malfunction is indicated, arrange for

service. Analyses may resume only when problem has been identified and corrected.

11. Repair and maintenance records are documented for inspection and review.
12. Analytical results including raw data and chromatographs of method blanks, three point standard calibration and quality control samples (matrix spikes and matrix spike duplicates) for the following methods are available for review:

Method Number	Description
632	Carbamates
8030	Acrolein, Acrylonitrile & Acetonitrile
8060	Phthalate Esters
8090	Nitroaromatics & Cyclic Ketones
8120	Chlorinated Hydrocarbons

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE LEVEL	ACCEPTABLE % RANGE	DUPLICATE ACCEPTABLE % DIFFERENCE
	Matrix	ppb		
Metals by AA *	Liquid	10X	80-115	15
	Solid		65-130	30
As	Liquid	468	70-120	25
	Solid	12500	60-135	35
Cd	Liquid	90	80-122	25
	Solid	12500	60-135	35
Cr	Liquid	916	75-120	20
	Solid	20000	60-135	35
Co	Liquid	303	82-115	15
	Solid	12500	60-135	40
Cu	Liquid	110	84-117	15
	Solid	180000	65-130	40
Pb	Liquid	556	85-120	15
	Solid	12000	75-125	25
Hg	Liquid	24	65-133	30
	Solid	6100	40-150	45
Ni	Liquid	121	82-118	15
	Solid	9000	55-140	40
Se	Liquid	45	50-131	40
	Solid	12500	40-150	45
V	Liquid	791	82-123	25
	Solid	9000	55-140	40
Zn	Liquid	1378	85-114	15
	Solid	360000	40-150	45
Tl	Solid	1800	50-145	40
Ag	Solid	1800	12-160	45
Sb	Solid	6100	60-135	35
Be	Solid	16300	55-140	40
Al	Liquid	815	78-125	25

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE

## SAMPLES-Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE		DUPLICATE
Matrix	LEVEL	ACCEPTABLE		ACCEPTABLE
	ppb	% RANGE		% DIFFERENCE
<b>Chlorinated</b>				
Herbicides *	Liquid	300	75-120	25
2,4-D	Liquid	300	75-120	25
Silvex	Liquid	70	75-120	25
2,4,5-T	Liquid	90	75-125	25
<b>Phenols *</b>				
	Liquid	300	40-140	45
	Solid			
2-Chlorophenol	Liquid	100	25-135	45
	Solid	10030	32-140	50
2-Nitrophenol	Liquid	500	D-150	100
	Solid	4020	D-150	100
Phenol	Liquid	300	5-112	75
	Solid	4000	D-150	100
2,4-Dimethyl-phenol	Liquid	300	12-150	75
	Solid	4020	D-150	100
2,4-Dichloro-phenol	Liquid	300	35-140	50
	Solid	10100	19-150	75
2,4,6-Tri-chlorophenol	Liquid	300	20-150	75
	Solid	10020	20-150	75
4-Chloro-3-methylphenol	Liquid	200	22-147	75
	Solid	10020	15-150	75
2,4-Dinitro-phenol	Liquid	500	D-160	100
	Solid	10000	D-150	100
2-Methyl-4,6-dinitrophenol	Liquid	500	5-160	75
	Solid	4020	D-160	100
Pentachloro-phenol	Liquid	100	14-176	75
	Solid	4010	D-180	100
4-Nitrophenol	Liquid	500	D-132	75
	Solid	4010	D-160	100

- TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES

- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE LEVEL    ACCEPTABLE		DUPLICATE
	Matrix	ppb	% RANGE	ACCEPTABLE % DIFFERENCE
-----				
Metals by ICP *	Liquid	10X	50-140	40
As	Liquid	22	70-125	25
Cd	Liquid	2.5	52-140	40
Cr	Liquid	10	46-145	40
Cu	Liquid	11	10-160	75
Pb	Liquid	24	15-155	75
Ni	Liquid	60	58-135	40
Se	Liquid	6	D-200	100
Zn	Liquid	16	10-170	75
Be	Liquid	20	70-125	25
Co	Liquid	120	40-150	50
V	Liquid	70	80-115	15
Carbamate by LC	Liquid	100X	80-115	15
Polychlorinated Biphenyls (see organochlorine pesticides)				
Aromatic Volatile Organics				
Benzene	Liquid	9	64-130	40
Chlorobenzene	Liquid	100	70-127	30
1,2-Dichloro- benzene	Liquid	10	16-170	75
1,3-Dichloro- benzene	Liquid	5	37-150	75
1,4-Dichloro- benzene	Liquid	10	40-170	75
Ethylbenzene	Liquid	10	62-137	40
Toluene	Liquid	100	41-136	75

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES  
- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE LEVEL ACCEPTABLE		DUPLICATE
Matrix		ppb	% RANGE	ACCEPTABLE % DIFFERENCE
-----				
Halogenated Volatile Organics				
Bromodichloro-				
methane	Liquid	0.5	85-115	15
Bromoform	Liquid	50	60-130	30
Carbon				
tetrachloride	Liquid	50	20-140	75
Chlorobenzene	Liquid	50	67-121	25
Chloroethane	Liquid	50	30-150	75
2-Chloroethyl				
vinyl ether	Liquid	133	66-126	30
Chloroform	Liquid	50	60-160	40
Chloromethane	Liquid	24	50-140	50
Dibromochloro-				
methane	Liquid	93	78-118	25
1,2-Dichloro-				
benzene	Liquid	150	85-115	15
1,3-Dichloro-				
benzene	Liquid	50	79-112	25
1,4-Dichloro-				
benzene	Liquid	50	70-124	25
1,1-Dichloro-				
ethane	Liquid	45	80-120	25
1,2-Dichloro-				
ethane	Liquid	45	80-120	25
1,1-Dichloro-				
ethylene	Liquid	50	40-150	60
t-1,2-Dichloro-				
ethylene	Liquid	98	30-150	75
1,2-Dichloro-				
propane	Liquid	39	70-125	25
t-1,3-Dichloro-				
propylene	Liquid	50	22-150	75
1,1,2,2-Tetra-				
chloroethane	Liquid	45	45-135	45
Tetrachloro-				
ethylene	Liquid	35	35-150	75
1,1,1-Tri-				
chloroethane	Liquid	29	45-135	50
1,1,2-Tri-				
chloroethane	Liquid	50	20-150	75
Trichloro-				
ethylene	Liquid	45	80-120	25
Vinyl Chloride	Liquid	32	70-130	25

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES  
- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE		DUPLICATE
Matrix		LEVEL	ACCEPTABLE	ACCEPTABLE
		ppb	% RANGE	% DIFFERENCE
-----				
Base/Neutrals				
Acenaphthene	Liquid	100	47-145	45
	Solid	2010	26-130	75
Acenaphthalene	Liquid	2000	31-140	75
	Solid	2020	25-130	75
Anthracene	Liquid	2000	30-140	75
	Solid	4040	32-132	75
Benzo(a)anthracene	Liquid	2000	30-140	75
	Solid	2020	7-150	100
Benzo(b)fluoranthene	Liquid	2000	17-140	100
	Solid	4420	6-150	100
Benzo(k)fluoranthene	Liquid	2000	17-140	100
	Solid	4420	6-150	100
Benzo(ghi)perylene	Liquid	2000	D-180	200
	Solid	62000	40-160	75
Benzo(a)pyrene	Liquid	2000	17-140	100
	Solid	2080	18-130	100
Benzidine	Liquid	2000	0-150	200
	Solid	2010	EPA NOT ABLE TO RECOVER	
Butyl benzyl phthalate	Liquid	2000	D-150	200
	Solid	2020	7-150	100
Bis(2-chloroethoxy)methane	Liquid	2000	D-150	200
	Solid	2020	9-150	100
Bis(2-chloroethyl)ether	Liquid	2000	D-150	200
	Solid	2020	D-150	200
Bis(2-chloroisopropyl)ether	Liquid	2000	D-150	200
	Solid	10020	30-150	75
Bis(2-ethylhexyl)phthal.	Liquid	2000	D-200	200
	Solid	32210	30-150	75
4-Bromophenyl phenyl ether	Liquid	2000	30-150	75
	Solid	2070	30-150	75
2-Chloronaphthalene	Liquid	2000	37-150	75
	Solid	1920	30-150	75
4-chlorophenyl phenyl ether	Liquid	2000	30-150	75
	Solid	2010	15-130	100
Chrysene	Liquid	2000	37-150	75
	Solid	2020	7-150	100
Dibenzo(a,h)anthracene	Liquid	2000	D-170	200
	Solid	61400	34-200	100
Di-n-butyl phthalate	Liquid	2000	D-200	200
	Solid	2010	5-180	100
1,2-Dichlorobenzene	Liquid	100	1-118	125
	Solid	77200	50-128	50

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES  
- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE LEVEL ACCEPTABLE		DUPLICATE
Matrix		ppb	% RANGE	ACCEPTABLE % DIFFERENCE
1,3-Dichloro	Liquid	100	1-118	125
benzene	Solid	8540	D-170	200
1,3-Dichloro	Liquid	100	1-118	125
benzene	Solid	54800	29-142	75
3,3-Dichloro-	Liquid	2000	D-300	200
benzidine	Solid	10000	D-300	200
Dimethyl	Liquid	2000	3-130	125
phthalate	Solid	2010	21-140	100
2,4-Dinitro-	Liquid	200	50-158	75
toluene	Solid	2030	18-140	100
2,6-Dinitro-	Liquid	200	50-158	75
toluene	Solid	2030	18-140	100
Di-n-octyl	Liquid	2000	D-200	200
phthalate	Solid	2030	12-170	100
Fluoranthene	Liquid	2000	20-140	100
	Solid	2010	D-170	150
Fluorene	Liquid	2000	20-140	100
	Solid	2010	30-160	100
Hexachloro-	Liquid	2000	26-150	100
benzene	Solid	1610	63-133	50
Hexachloro-	Liquid	2000	D-200	200
butadiene	Solid	2050	0-200	200
Hexachloro-	Liquid	2000	D-200	200
cypentadiene	Solid	2010	EPA NOT ABLE TO RECOVER	
Hexachloro-	Liquid	2000	D-200	200
ethane	Solid	2000	56-151	75
Indeno(1,2,3-	Liquid	2000	D-200	200
cd) pyrene	Solid	50010	34-180	100
Isophorone	Liquid	2000	D-200	200
	Solid	2020	9-142	100
Naphthalene	Liquid	2000	D-200	200
	Solid	152500	55-126	75
Nitrobenzene	Liquid	2000	D-200	200
	Solid	42100	16-150	125
N-Nitrosodi-n-	Liquid	2000	D-200	200
propylamine	Solid	10020	62-180	75
N-Nitrosodi-	Liquid	2000	6-170	100
phenylamine	Solid	10000	0-200	200
Phenanthrene	Liquid	2000	29-136	100
	Solid	2010	26-130	75
Pyrene	Liquid	2000	29-136	100
	Solid	2020	16-160	100
Trichloro-	Liquid	2000	17-170	100
benzene	Solid	2200	3-160	125

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES  
- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE LEVEL	ACCEPTABLE % RANGE	DUPLICATE ACCEPTABLE % DIFFERENCE
Matrix		ppb		
EPA 624/8240 Purgeable Organics				
Benzene	Liquid	100	68-130	40
	Solid	99	80-100	50
Bromodichloro- methane	Liquid	100	73-130	30
	Solid	10	60-170	50
Bromoform	Liquid	100	52-152	50
	Solid	10	70-150	50
Bromomethane	Liquid	100	22-169	75
Carbon tetra- chloride	Liquid	100	69-149	40
	Solid	10	61-150	50
Chlorobenzene	Liquid	100	75-130	30
	Solid	10	10-150	100
Chloroethane	Liquid	100	14-190	100
2-Chloroethyl vinyl ether	Liquid	100	75-150	35
Chloroform	Liquid	100	64-136	40
	Solid	10	85-132	40
Chloromethane	Liquid	100	28-172	75
Dibromochloro- methane	Liquid	100	62-146	40
	Solid	1	33-126	75
1,1-Dichloro- ethane	Liquid	100	60-150	40
	Solid	10	31-150	75
1,2-Dichloro- ethane	Liquid	100	72-132	35
	Solid	10	13-160	100
1,1-Dichloro- ethene	Liquid	100	54-145	40
	Solid	10	10-170	100
1,2-Dichloro- ethene	Liquid	100	71-130	35
	Solid	10	23-147	100
1,2-Dichloro- propane	Liquid	100	67-136	40
	Solid	10	35-144	75
1,3-Dichloro- propene	Liquid	100	45-157	50
	Solid	8	45-140	75
1,3-Dichloro- propene	Liquid	100	46-154	50
	Solid	10	48-142	100
Ethylbenzene	Liquid	100	73-133	40
	Solid	10	43-130	100
Methylene Chloride	Liquid	100	8-187	200
	Solid	10	28-130	200
1,1,2,2-Tetra- chloroethane	Liquid	100	60-146	40
	Solid	10	D-250	300
Tetrachloro- ethene	Liquid	100	67-133	70
	Solid	10	D-250	300

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES  
- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE		DUPLICATE
	Matrix	LEVEL ppb	ACCEPTABLE % RANGE	ACCEPTABLE % DIFFERENCE
Toluene	Liquid	100	56-140	40
	Solid	10	61-120	45
1,1,1-Tri- chloroethane	Liquid	100	54-150	40
	Solid	10	23-140	150
1,1,2-Tri- chloroethane	Liquid	100	59-149	40
	Solid	10	55-127	50
Trichloro- ethene	Liquid	100	64-136	40
	Solid	10	18-145	150
Trichloro- fluoromethane	Liquid	100	50-164	75
Vinyl chloride	Liquid	100	23-173	180
EPA 8030 PURGEABLE ORGANIC COMPOUNDS				
Acrolein	Liquid	20	61-135	45
Acrylonitrile	Liquid	20	85-124	40
EPA 8060 PHTHALATE ESTERS				
Bis(2-ethyl- hexyl)phthalate	Liquid	1000	74-111	45
Butyl benzyl phthalate	Liquid	1000	66-116	50
Dibutyl- phthalate	Liquid	1000	65-115	50
Diethyl phthalate	Liquid	50	90-110	40
Dimethyl phthalate	Liquid	50	84-110	40
Dioctyl phthalate	Liquid	150	73-113	40
EPA 8090 NITROAROMATICS AND CYCLIC KETONES				
2,4-Dinitro- toluene	Liquid	100	55-110	75
2,6-Dinitro- Isophorone	Liquid	50	60-110	75
toluene	Liquid	50	63-110	75
Nitrobenzene	Liquid	100	58-113	75

EPA 8100 POLYNUCLEAR AROMATIC HYDROCARBONS

Same as EPA 625/8270

TABLE I - ACCEPTANCE LIMITS for QUALITY ASSURANCE SAMPLES  
- Continued

ANALYTICAL PROCEDURE or PARAMETER GROUP		SPIKE LEVEL ACCEPTABLE		DUPLICATE
Matrix		ppb	% RANGE	ACCEPTABLE % DIFFERENCE
EPA 8120 CHLORINATED HYDROCARBONS				
2-Chloro-naphthalene	Liquid	200	19-157	180
1,2-Dichloro-benzene	Liquid	300	57-125	100
1,3-Dichloro-benzene	Liquid	200	40-146	150
1,4-Dichloro-benzene	Liquid	300	35-153	150
Hexachloro-benzene	Liquid	10	61-134	75
Hexachloro-butadiene	Liquid	30	66-130	75
Hexachloro-ethane	Liquid	10	63-136	75
1,2,4-Tri-chlorobenzene	Liquid	10	48-148	125
EPA 8140 ORGANOPHOSPORUS PESTICIDES				
Azinphos methyl	Liquid	200	32-141	150
Bolstar	Liquid	40	53-115	75
Chlorpyrifos	Liquid	50	82-116	45
Coumaphos	Liquid	200	70-147	75
Demeton	Liquid	300	46-121	100
Diazinon	Liquid	5	55-112	75
Dichlorvos	Liquid	500	55-117	75
Disulfoton	Liquid	80	60-122	75
Ethoprop	Liquid	50	88-112	45
Fensulfothion	Liquid	100	46-148	150
Fenthion	Liquid	50	33-136	150
Merphos	Liquid	50	76-145	75
Mevinphos	Liquid	500	34-123	150
Naled	Liquid	200	69-119	75
Parathion methyl	Liquid	500	81-115	45
Phorate	Liquid	40	46-117	150
Ronnel	Liquid	50	83-117	45
Stirophos	Liquid	500	54-112	150
Tokuthion	Liquid	50	51-113	150
Trichloronate	Liquid	20	46-161	150

93, 96-98, and 104-115 to new Table IB, entitled "List of Approved Inorganic Test Procedures", adding two new inorganic parameters, Carbonaceous Bimolecular Oxygen Demand (CBOD<sub>k</sub>) and Nitrate-Nitrite, including an additional test procedure based upon the inductively coupled plasma technique in Table IB for 25 of the metal parameter designations, by including 10 methods approved under the equivalency provisions of §§ 136.4(d) and 136.5(e), and updating references to EPA, Standard Methods, ASTM, AOAC and USGS test procedures; by deleting

former parameter 14 (Chlorinated organic compounds) and by entering the individual chlorinated organic compounds into new Table IC, entitled, "List of Approved Test Procedures for Non-Pesticide Organic Compounds", transferring old parameters 9 (Benzidine) and 94 (Pentachlorophenol) to Table IC, by including the 78 additional proposed non-pesticidal organic parameters and by adding 17 new test procedures in Table IC; by deleting former parameter 95 (Pesticides) and by entering the 68 individual pesticides into new Table ID,

entitled "List of Approved Test Procedures for Pesticides", by including the 2 additional proposed pesticide parameters, and the two new test procedures in Table IB; and by transferring the former radiological parameters 99-103 to new Table IF, entitled "Approved Radiological Test Procedures", adding an EPA reference to the approved test procedures, and updating the Standard Methods, ASTM and USGS references. As revised, Table I reads as follows:

### § 136.3 Identification of Test Procedures.

TABLE IA.—LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter and units	Method <sup>1</sup>	EPA <sup>2</sup>	Reference (Method Number or Page)		
			Standard Methods 15th Ed.	ASTM	USGS
<b>Bacteria:</b>					
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution, or, membrane filter (MF) <sup>3</sup> , single step	p. 132	980C		B-0050-77.
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution	p. 124	908C		
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF <sup>3</sup> single step or two step	p. 132	908C		
		p. 114	908A		B-0025-77.
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or MF <sup>3</sup> with enrichment	p. 109	908A		
		p. 114	908A		
		p. 111	908 (A-A.5c)		
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF <sup>3</sup> ; or, plate count	p. 139	910A		B0055-77. <sup>4</sup>
		p. 136			
		p. 143			

**Table IA Notes**

<sup>1</sup> The method must be specified when results are reported

<sup>2</sup> "Microbiological Methods for Monitoring the Environment, Water and Wastes, 1978", EPA-600/8-78-017, U.S. Environmental Protection Agency.

<sup>3</sup> Green, P.E., et al., Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, "U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A4, Laboratory Analysis, 1977

<sup>4</sup> 0.45 um membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extraneous which could interfere with their growth and development.

<sup>5</sup> Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method B-0055-77) is made in a boiling water bath to avoid scorching of the medium.

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units, and method	Reference (method No. or page)				
	EPA 1979	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
1. Acidity, as CaCO <sub>3</sub> , mg/L. Electrometric and point or phenolphthalein end point	305.1	402(4.6)	D1067-70(E)		
2. Alkalinity, as CaCO <sub>3</sub> , mg/L. Electrometric or colorimetric					
Titration to pH 4.5, manual	310.1	403	D1067(B)	1-1808-78	P. 548. <sup>2</sup>
Or automated	310.2			1-2826-78	
3. Aluminum—Total <sup>3</sup> , mg/L. Digestion <sup>3</sup> followed by:					
AA direct aspiration	202.1	303C		1-3894-78	Method 200.7. <sup>4</sup>
AA furnace	202.2	304			
Inductively coupled plasma					
Or colorimetric (Eriochrome cyanine R)		306B			
4. Ammonia (as N), mg/L. Manual distillation <sup>5</sup> (at pH 9.5):					
Followed by	350.2	417A			P. 553. <sup>2</sup>
Nesslerization	350.2	417B	D1426-78(A)	1-3526-78	
Titration	350.2	417D			
Electrode	350.3		D1426-78(D)		
Automated phenate, or	350.1	417F	D1426-78(C)	1-4523-78	
Automated electrode					
5. Arsenic—Total <sup>3</sup> , mg/L. Digestion <sup>3</sup> followed by:					
AA direct aspiration	204.1	303A			Method 200.7. <sup>4</sup>
AA furnace, or	204.2	304			
Inductively coupled plasma					
Or, colorimetric (SDOC)	206.4	307B	D2972-78(A)	1-3088-78	
6. Barium—Total <sup>3</sup> , mg/L. Digestion <sup>3</sup> followed by:					
Hydride	206.5	303E	D2972-78(B)	1-3082-78	Method 200.7. <sup>4</sup>
AA furnace	206.3	304			
Inductively coupled plasma					
Or, colorimetric (SDOC)	206.4	307B	D2972-78(A)	1-3088-78	
7. Barium—Total <sup>3</sup> , mg/L. Digestion <sup>3</sup> followed by:					
AA direct aspiration	208.1	303C		1-3084-78	Method 200.7. <sup>4</sup>
AA furnace, or	208.2	304			
Inductively coupled plasma					
8. Beryllium—Total <sup>3</sup> , mg/L. Digestion <sup>3</sup> followed by:					
AA direct aspiration	210.1	303C	D3645-78	1-3085-78	
AA furnace	210.2	304			

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units, and method	Reference (method No. or page)				
	EPA 1979	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
Inductively coupled plasma Or colorimetric (aluminum)		309B			Method 200.7. <sup>a</sup>
9. Biochemical oxygen demand (BOD <sub>5</sub> ), mg/L: Winkler (Azide modification) Or electrode method	405.1	507		I-1578-78	P. 17. <sup>a</sup> P. 548. <sup>a</sup>
10. Boron—Total, mg/L: Colorimetric (curcumin) or Inductively coupled plasma	212.3	404A		I-3112-78	
11. Bromide, mg/L: Titrimetric	320.1		D1248-77(C)	I-1125-78	Method 200.7. <sup>a</sup> P. 844. <sup>10</sup>
12. Cadmium—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace Inductively coupled plasma Voltammetry <sup>4</sup> or Colorimetric (dimethylglyoxime)	213.2 213.2   310B	303A or 303B 304	D3557-78 (A or B)	I-3135-78 or I-3136-78	Pg. 557. <sup>a</sup> P. 37. <sup>a</sup> Method 200.7. <sup>a</sup>
13. Calcium—Total <sup>5</sup> , mg/L: Digestion <sup>3</sup> followed by: Atomic absorption Inductively coupled plasma Or EDTA titration	215.1 215.2	303A 311C	D511-77(C) D511-77(B)	D152-78	Method 200.7. <sup>a</sup>
14. Carbonaceous Biochemical oxygen demand (CBOD <sub>5</sub> ), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor.		507(5.e.f.)			
15. Chemical oxygen demand (COD), mg/L: Titrimetric colorimetric Manual or Automated Spectrophotometric	410.1 410.2 410.3 410.4	506A	D1252-78	I-3560-78 I-3562-78 I-3561-78	P. 550 <sup>a</sup> and P. 17 <sup>a</sup> and (1 <sup>10</sup> )
16. Chloride, mg/L: Titrimetric (silver nitrate) or Mercuric nitrate Colorimetric (mercuric cyanide) manual or Automated	325.3 325.1 or 325.2	407A 407B 407D	D512-67(B) D512-67(A) D512-67(C)	I-1183-78 I-1184-78 I-1187-78 I-2187-78	P. 554. <sup>a</sup>
17. Chlorine—Total residual, mg/L: Titrimetric-mercurimetric <sup>11</sup> Starch end point Iodometric or DPD-FAS Spectrophotometric, DPD, or Electrode	330.1 330.2 330.3 330.4 330.5	408C 408B 408A 408D 408E	D1253-76(A) D1253-76(B)		(1 <sup>11</sup> )
18. Chromium VI dissolved, mg/L: 0.45 micron titration with: Extraction and atomic absorption, or Colorimetric (Diphenylcarbazide)	218.4	303B		I-1232-78 I-1230-78	(1 <sup>12</sup> )
19. Chromium—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> (optional extraction) followed by: AA direct aspiration AA furnace Inductively coupled plasma Or colorimetric (Diphenylcarbazide)	218.3 218.1 218.2	303A or 303B 304	D1687-77(D) D1687-77(A)	I-3236-78	P. 567. <sup>a</sup> Method 200.7. <sup>a</sup>
20. Cobalt—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace, or Inductively coupled plasma	219.1 219.2	303A or 303B 304	D3558-77 (A or B)	I-3240-78 or I-3238-78	P. 37. <sup>a</sup> Method 200.7. <sup>a</sup>
21. Color, platinum Cobalt units or dominant wavelength hue, luminance, purity Colorimetric, ADM Platinum cobalt, or Spectrophotometric	110.1 110.2 110.3	204D 204A 204B		I-1250-78	(1 <sup>13</sup> )
22. Copper—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace Inductively coupled plasma Colorimetric (Neocupron) Bismuthimetric	220.1 220.2	303A or 303B 304	D1688-77 (D or E)	I-3271-78 or I-3270-78	P. 557 <sup>a</sup> and P. 37. <sup>a</sup> Method 200.7. <sup>a</sup>
23. Cyanide—Total mg/L: Manual distillation with MgO, Followed by titrimetric Manual or Automated <sup>14</sup> spectrophotometric	335.2 335.2 335.2 335.3	412D 412B 412C 412D	D2006-75(A) D2006-75(A) D2006-75(B)	I-3300-78	P. 22. <sup>a</sup>
24. Cyanide convertible to chloroform, mg/L: Manual distillation with MgO, Followed by titrimetric, manual or automated <sup>14</sup> spectrophotometric	335.1	412F			
25. Fluoride—Total, mg/L: Manual distillation <sup>15</sup> Followed by manual or Automated electrode SPADNS <sup>16</sup> Or automated complexone <sup>17</sup>	340.7 340.1 340.3	413A 413B 413C 413E	D1179-72(B) D1179-72(A)	I-4327-78	
26. Gold—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration Or AA furnace	231.1 231.2	303A 304			
27. Hardness—Total as CaCO <sub>3</sub> , mg/L: Automated colorimetric EDTA titration Inductively coupled plasma Or atomic absorption (sum of Ca and Mg as their respective carbonates)	130.1 130.2 215.1 242.1	314B 303A	D1125-67(B)	I-1338-78 I-3153-78 I-3448-78	Method 200.7. <sup>a</sup>

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units, and method	Reference (method No. or page)				
	EPA 1079	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
28. Hydrogen ion (pH), pH units: Electrometric	150.1	423	D1293-78(A) or D1293-78(B).	1-1586-78	( <sup>m</sup> )
Measurements; or automated electrode					
29. Indium—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	235.1	303A			
Or AA furnace	235.2	304			
30. Iron—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	236.1	303A or 303B	D1068-77 (C or D)	1-3381-78	P. 557. <sup>9</sup>
AA furnace	236.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					( <sup>m</sup> )
Or colorimetric (Phenanthroline)		315B	D1068-77(A)		P. 552. <sup>9</sup>
31. Kjeldahl nitrogen—Total (as N), mg/L: Digestion and distillation	351.3	420A or B			
Followed by titration	351.3	417D	D3590-77		
Neutralization or	351.3	417B			
Electrode	351.3	417E		1-4554-78	
Automated phenate	351.1			1-4553-78	
Semi-automated block digester	351.2				
Or potentiometric	351.4				
32. Lead—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	238.1	303A or 303B	D3558-78 (A or B)	1-3388-78	P. 557. <sup>9</sup>
AA furnace	238.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Voltammetry <sup>5</sup> or			D3558-78(C)		
Colorimetric (Dithione)		316B			
33. Magnesium—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: Atomic absorption	242.1	303A	D511-77(B)	1-3447-78	P. 557. <sup>9</sup>
Inductively coupled plasma					Method 200.7. <sup>4</sup>
Or gravimetric		318B	D511-77(A)		
34. Manganese—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	243.1	303A or 303B	D658-77 (B or C)	1-3454-78	P. 557. <sup>9</sup>
AA furnace	243.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					P. 564. <sup>9</sup>
Or colorimetric (Persulfate)		319B	D658-77(A)		18.
Periodate					
35. Mercury—Total <sup>2</sup> , mg/L: Cold vapor, manual or	245.1	303F	D3223-78	1-3483-78	P. 558. <sup>9</sup>
Automated	245.2				
36. Molybdenum—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	246.1	303C		1-3488-78	
AA furnace, or	246.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
37. Nickel—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	248.1	303A or 303B	D1886-77 (C or D)	1-3488-78	
AA furnace	248.2	304			Method 200.7. <sup>4</sup>
Inductively coupled plasma					
Or colorimetric (Mepytosine)		321B			
38. Nitrate (as N), mg/L: Bicine sulfate, or	352.1		D082-71		P. 564. <sup>9</sup>
Nitrate-nitrite N minus Nitrite N	See parameters 39 and 40.	See parameters 39 and 40.	See parameters 39 and 40.	See parameters 39 and 40.	P. 28. <sup>9</sup>
39. Nitrate-nitrite (as N), mg/L: Cadmium reduction, manual	353.3	418C	D3867-79(B)		
Or automated; or	353.2	418F	D3867-79(A)	1-4548-78	
Automated hydrazine	353.1				
40. Nitrite (as N), mg/L: Spectrophotometric, manual or	354.1	419	D1254-67		18.
Automated (Diazotization)				1-4548-78	
41. Oil and grease—Total recoverable, mg/L: Gravimetric (extraction)	413.1	503A			
42. Organic carbon—Total (TOC), mg/L: Combustion or oxidation	415.1	505	D2579-78(A) or D2579-78(B)		P. 561. <sup>9</sup> and P. 4. <sup>10</sup>
43. Organic nitrogen (as N), mg/L: Total Kjeldahl N minus ammonia N	See parameters 31 and 4.	420A	D3590-77 minus D1435-78(A)	See parameters 31 and 4.	PP. 562-63. <sup>9</sup>
44. Orthophosphate (as P), mg/L: Ascorbic acid method, automated	385.1	424G		1-4894-78	P. 561. <sup>9</sup>
Or manual single reagent or	385.2	424F	D515-78(A)		
Manual two reagent	385.3				
45. Cadmium—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration, or	252.1	303C			
AA furnace	252.2	304			
46. Oxygen, dissolved, mg/L: Winkler (Azide modification)	388.2	421B	D1589-80(A)	1-4575-78	P. 560. <sup>9</sup>
Or electrode	389.1	421F		1-4576-78	
47. Palladium—Total <sup>2</sup> , mg/L: Digestion <sup>3</sup> followed by: AA direct aspiration	253.1				P. 527. <sup>10</sup>
Or AA furnace	253.2				P. 528. <sup>10</sup>
48. Phenols, mg/L: Manual distillation	420.1		D1783-70 (A or B)		20.
Followed by manual	420.1				26.
Or automated <sup>11</sup> colorimetric (4AAP)	420.2				
49. Phosphorus (elemental), mg/L: Gas-liquid chromatography					21.

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units, and method	Reference (method No. or page)				
	EPA 1978	Standard methods 15th Ed.	ASTM	USGS <sup>1</sup>	Other
50. Phosphorus—Total, mg/L. Persulfate digestion Followed by manual or Automated ascorbic acid Reduction, or semi-automated block digester	365.2 365.2 or 365.3 365.1 365.4	424C (H) 424F 424G	D515-78(A)	I-4800-78 I-4803-78	P. 561. <sup>a</sup>
51. Platinum—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration Or AA furnace	255.1 255.2	303A 304			
52. Potassium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: Atomic absorption Inductively coupled plasma Or flame photometric	258.1	303A 322B 209A	D1428-84(A)	I-3630-78 I-3750-78 I-1750-78 I-3765-78	P. 560. <sup>a</sup> Method 200.7. <sup>a</sup>
53. Residue—total, mg/L. Gravimetric, 103-105°C.	160.3	209B			
54. Residue—filterable, mg/L. Gravimetric, 180°C.	160.1	209B			
55. Residue—nonfilterable, (TSS), mg/L. Gravimetric, 103-105°C post washing of residue.	160.2	209B			
56. Residue—settlesable, mg/L. Volumetric (limbott cone) or gravimetric.	160.5	209F			
57. Residue—soluble, mg/L. Gravimetric, 550°C.	160.4	209E		I-3753-78	
58. Rhodium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration Or AA furnace	265.1 267.2	303A 304			
59. Ruthenium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration Or AA furnace	267.1 267.2	303A 304			
60. Selenium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA furnace Inductively coupled plasma Or hydride	270.2 270.3	304 303E	D3658-78	I-3667-78	Method 200.7. <sup>a</sup>
61. Silica—Dissolved, mg/L. 0.45 micron filtration. Followed by manual or Automated colorimetric (Molybdosilicate), or Inductively coupled plasma	370.1	425C	D658-68(B)	I-1700-78 I-2700-78	Method 200.7. <sup>a</sup>
62. Silver—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace, or Inductively coupled plasma	272.1 272.1	303A or 303B 304		I-3720-78	P. 567. <sup>a</sup> and p. 37. <sup>a</sup> Method 200.7. <sup>a</sup>
63. Sodium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: Atomic absorption Inductively coupled plasma Or flame photometric	273.1	303A		I-3735-78	P. 561. <sup>a</sup> Method 200.7. <sup>a</sup>
64. Specific conductance, mhos/cm. Wheatstone bridge.	120.1	285	D1125-77(A)	I-1780-78	P. 547. <sup>a</sup>
65. Sulfate (as SO <sub>4</sub> ), mg/L: Automated methylthymol blue Gravimetric, or Turbidimetric	375.2 375.3 375.4	426A or 426B 426C	D516-68(A) D516-68(B)	I-2822-78	PP. 562-63. <sup>a</sup>
66. Sulfate (as S), mg/L: Titrimetric (barium) or Colorimetric (methylene blue)	376.1 376.2	427D 427C		I-3840-78	
67. Sulfate (as SO <sub>4</sub> ), mg/L. Titrimetric (iodine iodate)	377.1	428F	D1338-78(C)		
68. Sulfuric acid, mg/L. Colorimetric (methylene blue)	425.1	512A	D2130-68(A)		
69. Temperature, °C. Thermometric	170.1	212			(17).
70. Thallium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace, or Inductively coupled plasma	279.1 279.2	303A 304			Method 200.7. <sup>a</sup>
71. Tin—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration or AA furnace	282.1 282.2	303A 304		I-3850-78	
72. Titanium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration or AA furnace	283.1 283.2	303C 384			
73. Turbidity, NTU. Nephelometric	180.1	214A	D1889-71	I-3880-78	
74. Vanadium—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace Inductively coupled plasma Or colorimetric (Galleic acid)	286.1 286.2	303C 304			Method 200.7. <sup>a</sup>
75. Zinc—Total <sup>2</sup> , mg/L. Digestion <sup>3</sup> followed by: AA direct aspiration AA furnace Inductively coupled plasma Or colorimetric (Zincin)	289.1 289.2	303A or 303B 304	D1881-77(D) D1881-77(C)	I-3800-78	P. 567. <sup>a</sup> P. 37. <sup>a</sup> Method 200.7. <sup>a</sup> 24.

Table IB Notes

<sup>1</sup> "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 78-678, or "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," NW Skougstad, et al. U.S. Geological Survey, Techniques of Water-Resources Investigation, Book 5, Chapter A1, 1978.

<sup>2</sup> "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 13th ed. (1960).

<sup>3</sup> For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979." One (§ 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acid (§ 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by the vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and tellurium require a modified digestion and in all cases the method write-up should be consulted for specific instructions and/or cautions.

Notes: If the digestion procedure for direct aspiration or graphite furnace atomic absorption analysis included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals, or digestion of the original sample solution for total metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample has a low COD and the filtrate meets the following criteria:

- is visibly transparent
- Has the perceptible odor, and
- is free of particulate or suspended matter following acidification.

The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

Manual distillation is not required if comparability data on representative effluent samples are on company file to show that the preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

<sup>10</sup> Automated Electrode Method, Industrial Method Number 378-75WE, dated February 18, 1978, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

<sup>11</sup> Carbonaceous biochemical oxygen demand (CBOD) must not be confused with the traditional BOD, test which measures "lost BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD parameter. A discharger whose permit requires reporting the traditional BOD, may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD, is required can the permittee report data obtained using the nitrification inhibitor.

<sup>12</sup> American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

<sup>13</sup> The use of normal and differential pulse voltage scans to increase sensitivity and resolution is acceptable.

<sup>14</sup> Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>15</sup> COD Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.

<sup>16</sup> The back titration method will be used to resolve controversy.

<sup>17</sup> National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

<sup>18</sup> Copper, Bismuthomate Method, Method 8508, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>19</sup> After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.03 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335, the buffer 8.2 should be replaced with the buffer 7.8 found in Method 335.2.

<sup>20</sup> Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

<sup>21</sup> Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>22</sup> Manganese, Periodate Oxidation Method, Method 8004, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, Colorado 80537.

<sup>23</sup> Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

<sup>24</sup> Gomatos, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3, p.4 (1972).

<sup>25</sup> R.F. Addison and R.G. Actman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421-426, 1970.

<sup>26</sup> Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the recommended method is satisfactory.

<sup>27</sup> Savants, H.H., Ficks, J.F., and Smoot, G.F., "Water Temperature-Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 1, Chapter D1, 1975.

<sup>28</sup> Zinc, Zincon Method, Method 8008, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, Colorado 80537.

<sup>29</sup> "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

<sup>30</sup> The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.

<sup>31</sup> ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter <sup>1</sup>	EPA Method Number <sup>2,3</sup>			Other
	GC	GC/MS	HPLC	
1. Acenaphthene	610	625, 1625	610	
2. Acenaphthylene	610	625, 1625	610	
3. Acrolein	603	*624, 1624		
4. Acrylonitrile	603	*624, 1624		
5. Anthracene	610	625, 1625	610	
6. Benzene	602	624, 1624		
7. Benzidine		*625, 1625	605	Note 3, p. 1;
8. Benzo[a]anthracene	610	625, 1625	610	
9. Benzo[a]pyrene	610	625, 1625	610	
10. Benzo[b]fluoranthene	610	625, 1625	610	
11. Benzo[k]fluoranthene	610	625, 1625	610	
12. Benzo[a]pyrene	610	625, 1625	610	
13. Benzyl Chloride				Note 3, p. 130; Note 6, p. 5102.
14. Benzyl Ethyl Phthalate	606	625, 1625		
15. Bis(2-chloroethoxy) methane	611	625, 1625		
16. Bis(2-chloroethyl) ether	611	625, 1625		
17. Bis(2-ethylhexyl) phthalate	606	625, 1625		
18. Bisnaphthylmethane	601	624, 1624		
19. Bromofluoromethane	601	624, 1624		
20. Bromomethane	601	624, 1624		
21. 4-Bromophenylphenyl ether	611	625, 1625		
22. Carbon tetrachloride	601	624, 1624		Note 3, p. 130;
23. 4-Chloro-3-methylphenol	604	625, 1625		
24. Chlorobenzene	601, 602	624, 1624		Note 3, p. 130;
25. Chloroethane	601	624, 1624		
26. 2-Chloroethylvinyl ether	601	624, 1624		
27. Chloroform	601	624, 1624		Note 3, p. 130;
28. Chloromethane	601	624, 1624		
29. 2-Chlorophenol	612	625, 1625		
30. 2-Chlorophenol	604	625, 1625		
31. 4-Chlorophenylphenyl ether	611	625, 1625		
32. Chloroacetylene	610	625, 1625	610	
33. Dichloroethane	610	625, 1625	610	
34. Dichloromethane	601	624, 1624		
35. 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625		
36. 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625		
37. 1,4-Dichlorobenzene	601, 602, 612	625, 1624, 1625		
38. 2,3-Dichlorobenzidine		625, 1625	606	
39. Dichlorodifluoromethane	601			
40. 1,1-Dichloroethane	601	624, 1624		
41. 1,2-Dichloroethane	601	624, 1624		
42. 1,1-Dichloroethene	601	624, 1624		
43. Isene-1,2-Dichloroethene	601	624, 1624		
44. 2,4-Dichlorophenol	604	625, 1625		
45. 1,2-Dichloropropane	601	624, 1624		
46. cis-1,3-Dichloropropene	601	624, 1624		
47. Isene-1,3-Dichloropropene	601	624, 1624		
48. Diethyl phthalate	606	625, 1625		

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter <sup>1</sup>	EPA Method Number <sup>1, 2</sup>			Other
	GC	GC/MS	HPLC	
48. 2,4-Dimethylphenol	804	625, 1625		
49. Dimethyl phthalate	806	625, 1625		
50. Di-n-butyl phthalate	806	625, 1625		
51. Di-n-octyl phthalate	806	625, 1625		
52. 2,4-Dinitrophenol	804	625, 1625		
53. 2,4-Dinitroethane	806	625, 1625		
54. 2,6-Dinitroethane	806	625, 1625		
55. Epichlorohydrin				Note 2, p. 130; Note 6, p. 5162.
57. Ethylbenzene	802	624, 1624		
58. Fluorobenzene	810	625, 1625	618	
59. Fluorene	810	625, 1625	618	
60. Hexachlorobenzene	812	625, 1625		
61. Hexachlorobutadiene	812	625, 1625		
62. Hexachlorocyclopentadiene	812	625, 1625		
63. Hexachloroethane	812	625, 1625		
64. Menthyl(1,2,3-cd)pyrene	810	625, 1625	618	
65. Naphthalene	808	625, 1625		
66. Methylene Chloride	801	624, 1624		Note 2, p. 130;
67. 2-Methyl-4,6-Dinitrophenol	804	625, 1625		
68. Naphthalene	810	625, 1625		
69. Nitrobenzene	808	625, 1625		
70. 3-Nitrophenol	804	625, 1625		
71. 4-Nitrophenol	804	625, 1625		
72. N-Nitrosodimethylamine	807	625, 1625		
73. N-Nitroso-n-propylamine	807	625, 1625		
74. N-Nitrosodiphenylamine	807	625, 1625		
75. 2,2-dichloro(1-chloropropane)	811	625, 1625		
76. PCB-9918	808		625	Note 2, p. 42;
77. PCB-1221	808		625	Note 2, p. 42;
78. PCB-1232	808		625	Note 2, p. 42;
79. PCB-1242	808		625	Note 2, p. 42;
80. PCB-1248	808		625	Note 2, p. 42;
81. PCB-1254	808		625	Note 2, p. 42;
82. PCB-1268	808		625	Note 2, p. 42;
83. Pentachlorophenol	804	625, 1625		Note 2, p. 140;
84. Phenanthrene	810	625, 1625	618	
85. Phenol	804	625, 1625		
86. Pyrene	810	625, 1625	618	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin		613		
88. 1,1,2,2-Tetrachloroethane	801	624, 1624		Note 2, p. 130;
89. Tetrachloroethane	801	624, 1624		Note 2, p. 130;
90. Toluene	802	624, 1624		
91. 1,2,4-Trichlorobenzene	812	625, 1625		Note 2, p. 130;
92. 1,1,1-Trichloroethane	801	624, 1624		
93. 1,1,2-Trichloroethane	801	624, 1624		Note 2, p. 130;
94. Trichloroethane	801	624, 1624		
95. Trichlorofluoromethane	801	624		
96. 2,4,6-Trichlorophenol	804	625, 1625		
97. Vinyl Chloride	801	624, 1624		

Table IC Notes

<sup>1</sup>All parameters are expressed in micrograms per liter (µg/L).

<sup>2</sup>The full text of Methods 801-813, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

<sup>3</sup>Methods for Benzidine; Chlorinated Organic Compounds; Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 808 or Method 1624.

<sup>4</sup>Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 805, 807, and 812, or Method 1625, are preferred methods for these compounds.

<sup>5</sup>625, Screening only.

<sup>6</sup>Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

<sup>7</sup>Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 801-813, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 804 and 625 and 100% for Methods 1624, and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

Notes.—These warning limits are promulgated as an "interim final action with a request for comments."

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES <sup>1</sup>

Parameter µg/L	Method	EPA <sup>2, 3</sup>	Standard Methods 15th Ed	ASTM	Other
1. Aldrin	GC	808	508A	D0086	Note 2, p. 7; Note 4, p. 38.
2. Atrazine	GC/MS	625			
3. Azinphos	TLC				Note 2, p. 83; Note 6, p. 5162.
4. Azinphos	GC				Note 2, p. 84; Note 6, p. 5162.
5. Azinphos	GC				Note 2, p. 83; Note 6, p. 5162.
6. Azinphos methyl	GC				Note 2, p. 83; Note 6, p. 5162.
7. Barban	TLC				Note 2, p. 25; Note 6, p. 5162.
8. BHC	GC	808	508A	D0086	Note 2, p. 104; Note 6, p. 5162.
9. BHC	GC/MS	625			Note 2, p. 7.
10. BHC	GC	808		D0086	
11. BHC	GC/MS	625			
12. BHC	GC	808		D0086	

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES<sup>1</sup>—Continued

Parameter µg/L	Method	EPA <sup>2</sup>	Standard Methods 15th Ed	ASTM	Other
11. $\gamma$ -BHC (Lindane)	GC/MS GC GC/MS GC TLC GC GC GC/MS TLC	625 608 625    608 625	508A       508A  508B 508A	D3086       D3086  D3086	Note 3, p. 7; Note 4, p. 30.          Note 3, p. 104; Note 6, p. S64. Note 3, p. 115; Note 4, p. 35. Note 3, p. 7; Note 4, p. 30.
12. Captaf	GC		508A		Note 3, p. 7.
13. Carbaryl	TLC				Note 3, p. 94; Note 6, p. S60.
14. Carbofenthothion	GC				Note 4, p. 30; Note 6, p. S73.
15. Chlordane	GC GC/MS TLC	608 625  608 625	508A   508A  508B 508A	D3086   D3086  D3086	Note 3, p. 7.   Note 3, p. 104; Note 6, p. S64. Note 3, p. 115; Note 4, p. 35. Note 3, p. 7; Note 4, p. 30.
16. Chlorophen	TLC				Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		508B		Note 3, p. 115; Note 4, p. 35.
18. 4,4'-DDD	GC GC/MS GC GC/MS GC	608 625 608 625 608	508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30.  Note 3, p. 7; Note 4, p. 30.
19. 4,4'-DDE	GC GC/MS GC GC/MS GC	608 625 608 625 608	508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30.  Note 3, p. 7; Note 4, p. 30.
20. 4,4'-DDT	GC/MS GC GC/MS GC	625 608 625 608	508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30.  Note 3, p. 7; Note 4, p. 30.
21. Dactacon-O	GC				Note 3, p. 25; Note 6, p. S51.
22. Dactacon-S	GC				Note 3, p. 25; Note 6, p. S51.
23. Dactacon	GC				Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
24. Dactacon	(M)				Note 3, p. 115.
25. Dichlorodithion	GC				Note 4, p. 30; Note 6, p. S73.
26. Dichlorodithion	GC		508A		Note 3, p. 7.
27. Dieldrin	GC			D3086	Note 3, p. 7.
28. Dieldrin	GC GC/MS GC GC GC/MS GC	608 625   608 625	508A    508A  508B 508A	    D3086  D3086	Note 3, p. 7; Note 4, p. 30.  Note 4, p. 30; Note 6, p. S73. Note 3, p. 104; Note 6, p. S64. Note 3, p. 104; Note 6, p. S64.
29. Dieldrin	GC				Note 3, p. 7.
30. Dieldrin	GC				Note 3, p. 7.
31. Dieldrin	TLC				Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC GC/MS GC GC/MS GC	608 625 608 625 608	508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7.  Note 3, p. 7.
33. Endosulfan II	GC/MS GC GC/MS GC GC/MS GC	625 608 625 608 625 608	508A  508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7.  Note 3, p. 7.
34. Endosulfan sulfate	GC GC/MS GC GC/MS GC	608 625 608 625 608	508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30.
35. Endrin	GC/MS GC GC/MS GC GC/MS GC	625 608 625 608 625 608	508A  508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30.
36. Endrin aldehyde	GC/MS GC TLC TLC GC GC/MS GC	625 608   608 625 608	508A    508A  508A	D3086    D3086  D3086	Note 4, p. 30; Note 6, p. S73. Note 3, p. 104; Note 6, p. S64. Note 3, p. 104; Note 6, p. S64. Note 3, p. 7; Note 4, p. 30.
37. Ethion	GC				Note 4, p. 30; Note 6, p. S73.
38. Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39. Fenuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC GC/MS GC GC/MS GC	608 625 608 625 608	508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30.  Note 3, p. 7; Note 4, p. 30.
41. Heptachlor epoxide	GC GC/MS GC GC GC/MS GC	608 625 608 625 608 625	508A  508A  508A  508A	D3086  D3086  D3086	Note 3, p. 7; Note 4, p. 30; Note 6, p. S73.
42. Isodrin	GC/MS GC TLC GC	625   608	508A   508A	D3086   D3086	Note 4, p. 30; Note 6, p. S73. Note 3, p. 104; Note 6, p. S64. Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
43. Lurex	GC				Note 3, p. 94; Note 6, p. S80.
44. Malathion	GC		508A		Note 3, p. 7; Note 4, p. 30.
45. Methocarb	TLC				Note 3, p. 94; Note 6, p. S80.
46. Methoxychlor	GC		508A	D3086	Note 3, p. 7; Note 4, p. 30.
47. Mestacarb	TLC				Note 3, p. 94; Note 6, p. S80.
48. Mirex	GC		508A		Note 3, p. 7.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
50. Monuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
51. Naluron	TLC				Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		508A		Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl	GC		508A		Note 3, p. 25.
54. PCNB	GC		508A		Note 3, p. 7.
55. Permethrin	GC			D3086	Note 3, p. 7.
56. Permethrin	GC				Note 3, p. 83; Note 6, p. S88.
57. Permethrin	GC				Note 3, p. 83; Note 6, p. S88.
58. Permethrin	GC				Note 3, p. 83; Note 6, p. S88.
59. Permethrin	TLC				Note 3, p. 104; Note 6, p. S64.
60. Propoxur	TLC				Note 3, p. 94; Note 6, p. S80.
61. Sebacic acid	TLC				Note 3, p. 83; Note 6, p. S88.
62. Sebacic acid	TLC				Note 3, p. 104; Note 6, p. S64.
63. Sebacic acid	GC				Note 3, p. 83; Note 6, p. S88.
64. Sebacic acid	GC		508A		Note 3, p. 7.
65. Sebacic acid	TLC				Note 3, p. 104; Note 6, p. S64.
66. ZAS-7	GC		508B		Note 3, p. 115; Note 4, p. 35.
67. ZAS-7P (Solvent)	GC		508B		Note 3, p. 115.
68. Terbufos	GC				Note 3, p. 83; Note 6, p. S88.
69. Terbufos	GC/MS GC	608 625	508A 508A	D3086	Note 3, p. 7; Note 4, p. 30.
70. Terbufos	GC		508A		Note 3, p. 7.

Table ID Notes

- <sup>1</sup> Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.
- <sup>2</sup> The full text of methods 608 and 625 are given in Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given in Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.
- <sup>3</sup> "Methods for Benzene, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.
- <sup>4</sup> "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).
- <sup>5</sup> The method may be extended to include  $\alpha$ -BHC,  $\beta$ -BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.
- <sup>6</sup> "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).
- <sup>7</sup> Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with

Method 808 or 5% of all samples analyzed with Method 825 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. Note.—These warning limits are promulgated as an "interim final action with a request for comments."

TABLE IE.—LIST OF APPROVED RADIOLOGICAL TEST PROCEDURES

Parameter and units	Methods	EPA <sup>1</sup>	Reference (method No. or page)		
			Standard Methods 15th Ed	ASTM	USGS <sup>2</sup>
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.9	703	D1943-85	pp. 75 and 78. <sup>3</sup>
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	703	D1943-85	p. 79.
3. Alpha-Counting error, pCi per liter	Proportional counter	900.9	703	D1980-85	pp. 75 and 78. <sup>3</sup>
4. Beta-Counting error, pCi per liter	Proportional counter	Appendix B	703	D1980-85	p. 79.
5. (a) Radium-Total, pCi per liter	Proportional counter	900.9	705	D2480-79	
(b) "Ra, pCi per liter	Scintillation counter	900.1	706	D2154-79	p. 81.

## Table IE Notes

<sup>1</sup> Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032 (1980 update), U.S. Environmental Protection Agency, August 1980.

<sup>2</sup> Fehrmann, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 78-177 (1976).

<sup>3</sup> The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the total.

5. In § 136.3, paragraph (a) is revised to show that the full text of approved test procedures have been incorporated by reference, into the regulation to read as follows:

## § 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, and IE. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, and IE. The references and the sources from which they are available are given in paragraph (b) of this section. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published

in the Federal Register. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, and IE, or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and sections 136.4 and 136.5 of this Part 136. Under certain circumstances (§§ 136.3 (b) or (c) or 40 CFR Part 401.13) other test procedures may be used that may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur

does not object to the use of such alternate test procedure.

6. In § 136.3, paragraphs (b) and (c) are redesignated as (c) and (d) and a new paragraph (b) is added to itemize the references which are "incorporated by reference" and to identify the sources from which they may be obtained. As added, the new paragraph (b) reads as follows:

## § 136.3 Identification of test procedures.

(b) The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, and IE are incorporated by reference into this regulation and may be obtained from the sources identified. All costs cited are subject to change and must be verified from the indicated sources.

## REFERENCES, SOURCES, AND COSTS

Table	Parameters	Reference, source and cost
IA—EPA	1-5	"Microbiological Methods for Monitoring the Environment, Water and Wastes," United States Environmental Protection Agency, EPA-600/9-79-017, 1978. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268
IB—Standard Methods	1-5	
IC—Standard Methods	1-10, 12-48, 50-75	
ID—Standard Methods	1, 8, 11, 12, 15, 17-39, 26, 28, 32, 33, 35, 40, 41, 44, 46, 48, 52-54, 64, 66, 67, 69, 70	Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation 15th Edition, 1981. Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20006. Cost: \$50.00 including the Supplement to the Fifteenth Edition.
IE—Standard Methods	1-5	
IB—Standard Methods	48	Ibid, 14th Edition
IC—Other (Standard Methods Supplement)	11, 47	
ID—Other (Standard Methods Supplement)	13, 56	
ID—Other (Standard Methods Supplement)	2-7, 13, 14, 16, 21-23, 25, 29-31, 37, 38, 39, 41-45, 47, 49, 50, 51, 56-63, 65, 68	"Selected Analytical Methods approved and Cited by the United States Environmental Protection Agency," Supplement to the 12th Edition of Standard Methods for the Examination of Water and Wastewater (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20006. Cost: Included with the 15th Edition of Standard Methods for the Examination of Water and Wastewater
IB—U.S. Geological Survey (USGS)	1, 2, 5	
IC—EPA	1-13, 15-48, 50-75	"Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," edited by P.E. Greeson, T.A. Etkin, G.A. Sum, B.W. Lum, and K.V. Slack. U.S. Geological Survey, Techniques of Water-Resources Investigation (USGS TWRI), Book 5, Chapter A4 (1977). Revised edition, 332 pages. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. (Authorized agent of the Superintendent of Documents, Government Printing Office.) Cost: \$8.25. Prices are subject to change.
IB—ASTM	1, 2, 4, 6, 8, 11-13, 15-17, 19, 20, 22-25, 27, 28, 30-35, 37-40, 42-44, 46, 48, 50, 52, 60, 61, 63-65, 67, 68, 73-75	"Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 United States Environmental Protection Agency, March, 1979. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency Cincinnati, Ohio 45268
ID—ASTM	1, 8-11, 15, 18-20, 27, 32, 33, 35, 40, 41, 46, 55, 69	"Annual Book of Standards, Part 31, Water", American Society for Testing and Materials, 1980. Available from: American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Cost: available from publisher
IE—ASTM	1-5	

## REFERENCES, SOURCES, AND COSTS—Continued

Table	Parameters	Reference, source and cost
IS—USGS	2, 3, 4, 6-13, 15, 16, 18-23, 25, 27, 28, 30-40, 43, 44, 46, 50, 52-55, 57, 60-66, 71, 73, 75.	"Methods for determination of inorganic substances in water and fluvial sediments," H.W. Skougstad and others, editors: USGS—TWRI Book 5, Chapter A1, 1979 \$10.00. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202 (Authorized agent of the Superintendent of Documents, Government Printing Office). Prices are subject to change.
IS—Other (AOAC)	2, 4, 8, 12, 15, 16, 19, 22, 30-35, 38, 42-44, 46, 50, 52, 62-65, 75.	Official Methods of Analysis of the Association of Official Analytical Chemists, methods manual, 12th Edition (1980). Price: \$78.00. Available from: The Association of Official Analytical Chemists, 1111 N. 19th St., Suite 210, Arlington, VA 22209.
IS—Other (ANSI)	9, 12, 15, 20, 22, 23, 38, 62, 75.	"American National Standard on Photographic Processing Effluents," April 2, 1975. Available from: American National Standards Institute, 1430 Broadway, New York, New York 10018.
IS—Other (EPA)	3, 5-8, 10, 12, 13, 19, 20, 22, 27, 30, 32-34, 36, 37, 42, 40, 43, 70, 74, 75.	The full text of the inductively coupled plasma optical emission spectroscopic test procedure, Method 700.7, is printed in Appendix C of this Part 136.
IS—Other	71	"An Investigation of Inorganic Phosphorus for Measurement of Mill Effluent and Heavy Water," M. A. J. Federal Institute No. 253, (November, 1971). Available from: National Council of the Paper Industry for Air and Waste Improvements, Inc., 201 Madison Avenue (not available from publisher).
IS—Other	4	Anonymous, Automated Electrode Method, Industrial Method Number 378-75WE, dated February 19, 1976, Technicon AutoAnalyzer II Method and price available from Technicon Industrial Systems, Tarrytown, New York 10581.
IS—Other	15	Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 388, Loveland, Colorado 80537.
IS—Other	15	OC Chemical Oxygen Demand Method, Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2990, College Station, Texas 77840.
IS—Other	17	ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-78, 1977. Method and price available from Orion Research Incorporated, 848 Memorial Drive, Cambridge, Massachusetts 02138.
IS—Other	22	Biochromate Method for Copper, Method 8506, Hach Handbook of Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 388, Loveland, Colorado 80537.
IS—Other	28	Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II Method and Price available from Technicon Industrial Systems, Tarrytown, New York 10581.
IS—Other	30	1, 10-Phenanthroline Method for Iron, Hach Method 8008, Method and price available from Hach Chemical Company, P.O. Box 388, Loveland, Colorado 80537.
IS—Other	34	Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and Price available from Hach Chemical Company, P.O. Box 388, Loveland, Colorado 80537.
IS—Other	40	Nitrite Nitrogen, Hach Method 8507, Method and price available from Hach Chemical Company, P.O. Box 388, Loveland, Colorado 80537.
IS—Other	75	Zinc Method for Zinc, Method 8009, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 388, Loveland, Colorado 80537.
IS—Other	49	"Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," by R.F. Addison and R.G. Achman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1979. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher.
IS—Other (USGS)	69	"Water temperature-influenced factors, field measurement, and data presentation," by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot: USGS—TWRI Book 1, Chapter D1, 1975. 65 pages, \$1.60. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Prices are subject to change.
IS—Other (USGS) ID—Other (USGS)	42 1, 11, 14, 17-20, 23, 25, 28, 29, 35, 37, 40-42, 44, 46, 52, 66, 69	"Methods for analysis of organic substances in water," by D. F. Goertz and Eugene Brown: USGS—TWRI Book 5, Chapter A3, 1972, 40 pages, \$3.00. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Prices are subject to change.
IC—EPA ID—EPA	1-12, 14-65, 67-67 1, 8-11, 18, 19, 20, 28, 32-36, 40, 41, 69	The full texts of Methods 601-613, 624, 625, 1624, and 1625 are printed in appendix A of this Part 136. The full text for determining the method detection limit when using the test procedures is given in Appendix B of this Part 136.
IC—Other (EPA) ID—Other	7, 13, 22, 24, 27, 54, 66, 76-83, 86, 88, 91, 93 1-8, 11-13, 15-24, 26, 28, 30-33, 35, 38-41, 43-54, 56-70	"Methods for Benzene, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.
E—EPA	1-8	"Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-800/4-80-032 (1980 Update), United States Environmental Protection Agency, 1980. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.
E—USGS	1-8	"Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," by M.J. Fishman and Eugene Brown, U.S. Geological Survey Open File Report 78-77 (1978) \$13.50. Available from: U.S. Geological Survey, Branch Distribution, 1200 South Eads Street, Arlington, VA 22202.

The full texts of all the test procedures cited are available for inspection at the Office of the Federal Register Information Center, Room 8301, 1110 L Street, N.W., Washington, D.C. 20408.

7. In section 136.3 a new paragraph (e) is added together with a new Table II entitled, "Table II, Required Containers, Preservation Techniques, and Holding Times," to read as follows:

§ 136.3 Identification of test procedures.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are

prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to

assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by the Regional Administrator to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental

Monitoring and Support Laboratory, the Regional Administrator may grant a variance applicable to the specific discharge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
<b>Table IA—Bacterial Tests:</b>			
1-4. Coliform, fecal and total	P, G	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours.
5. Fecal streptococci	P, G	do	Do.
<b>Table IB—Inorganic Tests:</b>			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	do	Do.
4. Ammonia	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	do	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH > 12, 0.6g ascorbic acid <sup>6</sup>	14 days. <sup>6</sup>
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO <sub>3</sub> to pH < 2, H <sub>2</sub> SO <sub>4</sub> to pH < 2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
<b>Metals:<sup>7</sup></b>			
18. Chromium VI	P, G	Cool, 4°C	24 hours.
35. Mercury	P, G	HNO <sub>3</sub> to pH < 2	28 days.
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except chromium VI and mercury.	P, G	do	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
42. Organic carbon	P, G	Cool, 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH < 2	Do.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately.
47. Windsor	do	Fix on site and store in dark	6 hours.
48. Phenols	G only	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	do	48 hours.
55. Residue, Nonfilterable (TSS)	P, G	do	7 days.
56. Residue, Settleable	P, G	do	48 hours.
57. Residue, volatile	P, G	do	7 days.
61. Silica	P	do	28 days.
64. Specific conductance	P, G	do	Do.
65. Sulfate	P, G	do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH > 9	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P, G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
<b>Table IC—Organic Tests<sup>8</sup></b>			
13, 18-20, 22, 24-26, 34-37, 39-43, 45-47, 56, 58, 66, 68, 69, 82-85, 87. Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	14 days.
6, 57, 80. Purgeable aromatic hydrocarbons	do	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , HCl to pH < 2	Do.
3, 4, Acroton and acrylonitrile	do	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , Adjust pH to 4-5 <sup>10</sup>	Do.
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols <sup>11</sup>	G, Teflon-lined cap	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction.
7, 38. Benzodioxines <sup>12</sup>	do	do	7 days until extraction.
14, 17, 48, 50-52. Phthalate esters <sup>13</sup>	do	Cool, 4°C	7 days until extraction, 40 days after extraction.
72-74. Nitrosamines <sup>14, 15</sup>	do	Cool, 4°C, store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	Do.
76-82. PCBs <sup>16</sup> acrylonitrile	do	Cool, 4°C	Do.
54, 55, 65, 68. Nitrosamines and isophorone <sup>17</sup>	do	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup> , store in dark.	Do.
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 66, 84, 86. Polynuclear aromatic hydrocarbons <sup>18</sup>	do	do	Do.
15, 16, 21, 31, 75. Halocarbon <sup>19</sup>	do	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	Do.
29, 35-37, 60-63, 81. Chlorinated hydrocarbons <sup>20</sup>	do	Cool, 4°C	Do.
87. TCDD <sup>21</sup>	do	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	Do.
<b>Table ID—Pesticides Tests:</b>			
1-70. Pesticides <sup>22</sup>	do	Cool, 4°C, pH 5-9 <sup>23</sup>	Do.
<b>Table IE—Radiological Tests</b>			
1-5. Alpha, beta and radium	P, G	HNO <sub>3</sub> to pH < 2	6 months.

<sup>1</sup> Polyethylene (P) or Glass (G)

Table II Notes

<sup>1</sup> Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

<sup>2</sup> When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater), Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.82 or greater), Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater), and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

<sup>3</sup> Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.

<sup>4</sup> Should only be used in the presence of residual chlorine.

<sup>5</sup> Maximum holding time is 74 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium waste powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

<sup>6</sup> Samples should be filtered immediately on-site before adding preservatives for dissolved metals.

<sup>7</sup> Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

<sup>8</sup> Sample receiving no pH adjustment must be analyzed within seven days of sampling.

<sup>9</sup> The pH adjustment is not required if corrosion will not be measured. Samples for corrosion receiving no pH adjustment must be analyzed within 3 days of sampling.

<sup>10</sup> When the extractable analysis of concern fall within a single chemical category, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 8-9; samples preserved in the manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnotes 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

<sup>11</sup> If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

<sup>12</sup> Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxygen-free) atmosphere.

<sup>13</sup> For the analysis of diphenylhydrazine, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and adjust pH to 7-10 with NaOH within 24 hours of sampling.

<sup>14</sup> The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

8. Appendices A, B, and C are added to Part 136 to read as follows:

#### APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER

##### Method 601—Purgeable Halocarbons

###### 1. Scope and Application

1.1 This method covers the determination of 29 purgeable halocarbons.

The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Bromodichloromethane	32101	75-27-4
Bromotrichloromethane	32104	75-25-2
Bromomethane	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethyl vinyl ether	34576	100-75-8
Chloroform	32108	67-68-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34506	95-50-1
1,3-Dichlorobenzene	34508	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Dichlorodifluoromethane	34688	75-71-8
1,1-Dichloroethane	34498	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethene	34501	75-35-4
trans-1,2-Dichloroethane	34548	156-60-6
1,2-Dichloropropane	34541	78-67-8
cis-1,2-Dichloropropane	34704	10061-01-6
trans-1,2-Dichloropropane	34688	10061-02-8
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34518	79-34-8
Tetrachloroethane	34475	127-18-4
1,1,1-Trichloroethane	34508	71-65-6
1,1,2-Trichloroethane	34511	78-00-8
Tetrachloroethene	36180	78-01-6
Tetrachlorofluoromethane	34498	75-68-4
Vinyl chloride	36715	75-01-4

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the

primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1) for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

###### 2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.<sup>1,2</sup>

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

###### 3. Interferences

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic

tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

###### 4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified \*\* for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

### 5. Apparatus and Materials

#### 5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel (Section 6.3.4), 7.7 cm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long x 0.1 in. ID stainless steel or glass, packed with 1% SP-1000 on Carbowax B (60/80 mesh) or

equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long x 0.1 in. ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120 mesh) or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric detector. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The electrolytic conductivity detector was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-μL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

### 6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrisorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials:

6.3.1 Coconut charcoal—6/10 mesh sieved to 25 mesh, Barnebey Cheney, CA-580-26 lot # M-2849 or equivalent.

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used

when the analyst handles high concentrations of such materials.

6.5.1 Place about 0.8 mL of methanol into a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquid—Using a 100 μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30 °C (bromomethane, chloromethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL vaired gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10 to -20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Quality control check sample concentrate—See Section 8.2.1.

### 7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow

rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

### 7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0  $\mu$ L of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25- $\mu$ L syringe with a 0.008 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 6.2. If not so stored, they must be discarded after 1 h.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15  $\mu$ g/mL of each internal standard compound. The addition of 10  $\mu$ L of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30  $\mu$ g/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10  $\mu$ L of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_p)(C_{in})}{(A_{in})(C_p)}$$

where:

$A_p$  = Response for the parameter to be measured.

$A_{in}$  = Response for the internal standard.

$C_{in}$  = Concentration of the internal standard.

$C_p$  = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios,  $A_p/A_{in}$ , vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.5.4.

Note: The large number of parameters in Table 2 present a substantial probability that one or more will not meet the calibration acceptance criteria when all parameters are analyzed.

7.5.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

### 8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a

modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10  $\mu$ g/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20  $\mu$ g/L of each parameter by adding 200  $\mu$ L of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ( $\bar{X}$ ) in  $\mu$ g/L and the standard deviation of the recovery (s) in  $\mu$ g/L for each parameter of interest using the four results.

8.2.5 For each parameter compare s and  $\bar{X}$  with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and  $\bar{X}$  for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual  $\bar{X}$  falls outside the range for accuracy, then the system performance is unacceptable for that parameter.

Note: The large number of parameters in Table 2 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance

criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

**8.2.6.1** Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

**8.2.6.2** Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

**8.3** The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

**8.3.1** The concentration of the spike in the sample should be determined as follows:

**8.3.1.1** If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

**8.3.1.2** If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

**8.3.2** Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 µL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as  $100(A-B)/T$ , where T is the known true value of the spike.

**8.3.3** Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.<sup>1</sup> If spiking was performed at a concentration lower than 20 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for X; (3) calculate the range for recovery at the spike concentration as  $100 X'/T \pm 2.44(100 S'/T)\%$ .

**8.3.4** If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check

standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

**8.4** If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note:** The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 2 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

**8.4.1** Prepare the QC check standard by adding 10 µL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

**8.4.2** Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P) as  $100(A/T)\%$ , where T is the true value of the standard concentration.

**8.4.3** Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

**8.5** As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery (P̄) and the standard deviation of the percent recovery (s<sub>p</sub>). Express the accuracy assessment as a percent recovery interval from  $\bar{P} - 2s_p$  to  $\bar{P} + 2s_p$ . If  $p = 90\%$  and  $s_p = 10\%$ , for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

**8.6** It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

**8.7** The analyst should monitor both the performance of the analytical system and the

effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.5, add a volume to give 750 µg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 ng/µL. Add 10 µL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

## 9. Sample Collection, Preservation, and Handling

**9.1** All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl<sub>2</sub>) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.<sup>2</sup> Field test kits are available for this purpose.

**9.2** Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

**9.3** All samples must be analyzed within 14 days of collection.<sup>3</sup>

## 10. Procedure

**10.1** Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

**10.2** Calibrate the system daily as described in Section 7.

**10.3** Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

**10.4** Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual

air while adjusting the sample volume to 5.0  $\mu\text{L}$ . Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0  $\mu\text{L}$  of the surrogate spiking solution (Section 8.7) and 10.0  $\mu\text{L}$  of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for  $11.0 \pm 0.1$  min at ambient temperature.

10.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to  $180^\circ\text{C}$  while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to  $30^\circ\text{C}$  (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of  $45^\circ\text{C}$ .

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, condition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at  $180^\circ\text{C}$ . After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

### 11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})}{(A_{is})(\text{RF})}$$

where:

$A_s$  = Response for the parameter to be measured.

$A_{is}$  = Response for the internal standard.

$C_{is}$  = Concentration of the internal standard.

11.2 Report results in  $\mu\text{g/L}$  without correction for recovery data. All QC data obtained should be reported with the sample results.

### 12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1 were obtained using reagent water. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL to  $1000 \times \text{MDL}$ . Direct aqueous injection techniques should be used to measure concentration levels above  $1000 \times \text{MDL}$ .

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 8.0 to  $500 \mu\text{g/L}$ . Single

operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

### References

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- "Method Validation Data for EPA Method 801." Memorandum from B. Potter, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, November 10, 1983.

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit ( $\mu\text{g/L}$ )
	Column 1	Column 2	
Chloroethane	1.50	5.28	0.08
Bromoethane	2.17	7.05	1.18
Dichlorodifluoromethane	2.62	nd	1.81
Vinyl chloride	2.87	5.28	0.18
Chloroethane	3.33	8.68	0.52
1,1-dichloroethane	5.25	10.1	0.25
1,1,1-trichloroethane	7.18	nd	nd
1,1-Dichloroethane	7.83	7.72	0.13
1,1-Dichloroethane	9.30	12.8	0.87
trans-1,2-Dichloroethane	10.1	9.38	0.10

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Chloroform	10.7	12.1	0.05
1,2-Dichloroethane	11.4	15.4	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
Carbon tetrachloride	13.0	14.4	0.12
Bromodichloromethane	13.7	14.6	0.10
1,2-Dichloropropane	14.9	16.6	0.04
cis-1,3-Dichloropropene	15.2	16.6	0.34
Trichloroethane	15.8	13.1	0.12
Dibromochloromethane	16.5	16.6	0.09
1,1,2-Trichloroethane	16.5	18.1	0.02
trans-1,3-Dichloropropene	16.5	18.0	0.20
2-Chloroethylvinyl ether	18.0	nd	0.13
Bromobenzene	19.2	19.2	0.20
1,1,2,2-Tetrachloroethane	21.6	nd	0.03
Tetrachloroethane	21.7	15.0	0.03
Chlorobenzene	24.2	18.8	0.25
1,3-Dichlorobenzene	34.0	22.4	0.32
1,2-Dichlorobenzene	34.9	23.5	0.15
1,4-Dichlorobenzene	35.4	22.3	0.24

Column 1 conditions: Carbowax 8 (80/80 mesh) coated with 1% SP-1000 packed in an 8 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45 °C for 3 min then programmed at 8 °C/min to 220 °C and held for 15 min.

Column 2 conditions: Porat-C (100/120 mesh) coated with n-octane packed in a 8 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50 °C for 3 min then programmed at 8 °C/min to 170 °C and held for 4 min.

nd = not determined.

TABLE 2.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 601 \*

Parameter	Range for Q (µg/L)	Limit for s (µg/L)	Range for R (µg/L)	Range for P, P <sub>r</sub> (%)
Bromodichloromethane	15.2-24.8	4.3	10.7-32.0	42-172
Bromobenzene	14.7-25.3	4.7	5.0-29.3	13-158
Bromomethane	11.7-26.3	7.6	3.4-24.5	D-144
Carbon tetrachloride	13.7-26.3	5.6	11.8-25.3	43-143
Chlorobenzene	14.4-25.8	5.0	10.2-27.4	38-159
Chloroethane	15.4-24.6	4.4	11.3-25.2	46-137
2-Chloroethylvinyl ether	12.0-28.0	6.3	4.5-35.5	14-188
Chloroform	15.0-25.0	4.5	12.4-24.0	49-133
Chloromethane	11.9-29.1	7.4	0-34.9	D-188
Dibromochloromethane	13.1-26.9	6.3	7.9-35.1	24-191
1,2-Dichlorobenzene	14.0-26.0	5.5	1.7-38.9	D-208
1,3-Dichlorobenzene	9.9-30.1	9.1	6.2-32.6	7-167
1,4-Dichlorobenzene	13.9-26.1	5.5	11.5-25.5	42-143
1,1-Dichloroethane	16.8-23.2	3.2	11.2-24.6	47-132
1,2-Dichloroethane	14.3-25.7	5.2	13.0-26.5	51-147
1,1-Dichloroethane	12.6-27.4	6.6	10.2-27.3	28-167
trans-1,2-Dichloroethane	12.8-27.2	6.4	11.4-27.1	38-155
1,2-Dichloropropane	14.8-25.2	5.2	10.1-29.9	44-156
cis-1,3-Dichloropropene	12.8-27.2	7.3	6.2-33.8	22-178
trans-1,3-Dichloropropene	12.8-27.2	7.3	6.2-33.8	22-178
Methylene chloride	15.5-24.5	4.0	7.0-27.6	25-162
1,1,2,2-Tetrachloroethane	9.8-30.2	9.2	6.6-31.8	8-184
Tetrachloroethane	14.0-26.0	5.4	8.1-29.6	26-162
1,1,1-Trichloroethane	14.2-25.8	4.9	10.8-24.8	41-138
1,1,2-Trichloroethane	15.7-24.3	3.9	9.8-25.4	38-138
Trichloroethane	15.4-24.6	4.2	9.2-26.6	35-146
Trichlorofluoromethane	13.3-26.7	6.0	7.4-28.1	21-156
Vinyl chloride	13.7-26.3	5.7	6.2-29.9	29-160

Q = Concentration measured in QC check sample, in µg/L (Section 7.5.3).

S = Standard deviation of low recovery measurements, in µg/L (Section 8.2.4).

R = Average recovery for low recovery measurements, in µg/L (Section 8.2.4).

P, P<sub>r</sub> = Percent recovery measured (Section 8.3.2, Section 8.4.2).

D = Detected; result must be greater than zero.

\* Citings were calculated assuming a QC check sample concentration of 20 µg/L.

Note: These criteria are based directly upon the method performance data in Table

3. Where necessary, the limits for recovery have been broadened to assure applicability

of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601

Parameter	Accuracy, as recovery, % (µg/L)	Single analyst precision, s, % (µg/L)	Overall precision, S, % (µg/L)
Bromodichloromethane	1.12C - 1.02	0.11R + 0.04	0.20R + 1.00
Bromobenzene	0.98C - 2.05	0.12R + 0.56	0.21R + 2.41
Bromomethane	0.76C - 1.27	0.26R + 0.27	0.36R + 0.94
Carbon tetrachloride	0.98C - 1.04	0.15R + 0.38	0.20R + 0.38
Chlorobenzene	1.00C - 1.23	0.15R - 0.02	0.18R + 1.21
Chloroethane	0.99C - 1.53	0.14R - 0.13	0.17R + 0.63
2-Chloroethylvinyl ether	1.00C	0.20R	0.35R
Chloroform	0.93C - 0.39	0.13R + 0.15	0.18R - 0.02
Chloromethane	0.77C - 0.18	0.28R - 0.31	0.52R + 1.31
Dibromochloromethane	0.94C - 2.72	0.11R + 1.19	0.24R + 1.88
1,2-Dichlorobenzene	0.93C - 1.70	0.20R + 0.97	0.13R + 6.13

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601—Continued

Parameter	Accuracy, as recovery, $\bar{X}$ ( $\mu\text{g/L}$ )	Single analyst precision, $s$ ( $\mu\text{g/L}$ )	Overall precision, $S$ ( $\mu\text{g/L}$ )
1,3-Dichlorobenzene	0.94% ± 0.43	0.14% ± 0.33	0.26% ± 0.34
1,4-Dichlorobenzene	0.93% ± 0.08	0.15% ± 0.76	0.20% ± 0.41
1,1-Dichloroethane	0.94% ± 1.08	0.08% ± 0.17	0.14% ± 0.04
1,2-Dichloroethane	1.04% ± 1.06	0.11% ± 0.76	0.15% ± 0.04
1,1-Dichloroethene	0.90% ± 0.67	0.21% ± 0.23	0.29% ± 0.40
trans-1,2-Dichloroethene	0.97% ± 0.16	0.11% ± 1.48	0.17% ± 1.46
1,2-Dichloropropane*	1.00%	0.12%	0.23%
cis-1,3-Dichloropropene*	1.00%	0.18%	0.32%
trans-1,3-Dichloropropene*	1.00%	0.18%	0.32%
Methylene chloride	0.91% ± 0.93	0.11% ± 0.33	0.21% ± 1.43
1,1,2,2-Tetrachloroethane	0.95% ± 0.19	0.14% ± 2.41	0.23% ± 2.79
Tetrachloroethene	0.94% ± 0.06	0.14% ± 0.36	0.18% ± 2.21
1,1,1-Trichloroethane	0.90% ± 0.16	0.15% ± 0.04	0.20% ± 0.37
1,1,2-Trichloroethane	0.86% ± 0.30	0.12% ± 0.14	0.19% ± 0.67
Trichloroethene	0.87% ± 0.48	0.12% ± 0.03	0.23% ± 0.30
Trichlorofluoroethene	0.89% ± 0.07	0.15% ± 0.67	0.26% ± 0.91
Vinyl chloride	0.97% ± 0.36	0.12% ± 0.65	0.27% ± 0.40

$\bar{X}$  = Expected recovery for one or more measurements of a sample containing a concentration of  $C$ , in  $\mu\text{g/L}$ .

$s$  = Expected single analyst standard deviation of measurements at an average concentration found of  $\bar{X}$ , in  $\mu\text{g/L}$ .

$S$  = Expected interlaboratory standard deviation of measurements at an average concentration found of  $\bar{X}$ , in  $\mu\text{g/L}$ .

$C$  = True value for the concentration, in  $\mu\text{g/L}$ .

$\bar{X}$  = Average recovery found for measurements of samples containing a concentration of  $C$ , in  $\mu\text{g/L}$ .

\* Estimates based upon the performance in a single laboratory.<sup>10</sup>

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